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INVESTIGATIONS LEADING TO THE DEVELOPMENT
OF A PRIMARY ZINC-SILVER OXIDE BATTERY
OF IMPROVED PERFORMANCE CHARACTERISTICS

FINAL REPORT

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TABLE OF CONTENTS

<u>Description</u>	<u>Page No.</u>
I. PURPOSE	1
II. ABSTRACT	2
III. FACTUAL DATA AND DISCUSSION	3
A. Separator Evaluation	3
1. General	3
2. Preliminary Design Study	4
a. Object	4
b. Procedure	4
3. Results	6
B. Negative Material Studies	8
1. Negative Material Density	8
a. Object	8
b. Procedure	8
c. Results and Conclusions	8
2. Zinc Plate Formation Investigation	9
a. Object	9
b. Procedure	9
c. Analysis and Discussion	14
C. Electroformation of the Silver Positive Plate	24
1. Object	24
2. General Procedure	24
a. First Factorial Experiment	25
b. Second Factorial Experiment	34
c. Third Factorial Experiment	36
d. Interrupted Charge Investigation	39
1) Procedure	39
2) Results	41

TABLE OF CONTENTS (CONT.)

<u>Description</u>	<u>Page No.</u>
D. Full Cell Studies	44
1. Ratio of Materials	44
a. Purpose	44
b. Procedure	44
c. Results	44
2. Electrolyte Additive Study	48
a. Purpose	48
b. Procedure	48
c. Results and Conclusions	49
3. Effect of Cell Tightness and Quantity of Electrolyte	49
a. Object	49
b. Procedure	49
c. Results and Conclusions	53
4. Prototype Cell Investigation	53
a. Cell Design	53
b. Test Procedure	53
c. Cell Capacities	56
IV. SUMMARY AND CONCLUSIONS	60
V. REFERENCES	66
VI. PERSONNEL	67
APPENDIX	68

I. PURPOSE

The purpose of this contract was to obtain basic design data which would allow the construction of a zinc silver oxide cell capable of activated stand periods and recharge abilities as follows:

- A. Stand period (or useful life) - thirty days
- B. Stand Temperature - 90°F
- C. Cycle Capability - six cycles in thirty days
 - 1. Five cycles removing 25% depth
 - 2. A final discharge of 100% capacity
- D. Battery voltage during discharge - 28 ± 2.0 volts
(1.40 ± 0.10 volts per cell)

II. ABSTRACT

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Development work has been directed toward the design of an improved silver oxide-zinc battery of limited cycle life. A specific goal has been thirty day activated stand at +90°F, during which time five partial discharges and one full discharge are conducted. Toward this end, phases of study have dealt with improved materials formulation, ratio of materials, electrolyte additives and effects of cell group "compactness" and volume of electrolyte. Whenever practical, the use of full or fractional designed experiments has been emphasized.

Approximately three hundred test cells have been subjected to cycle regimes of one month duration. A larger number of test cells were used in studies at improved material formulation. A prototype cell design including silver grid (both positive and negative), a decreased silver-zinc ratio, decreased material apparent densities, slightly less electrolyte and a more compact cell group has been evaluated. It is indicated that the cycle life goal has been attained, along with an increase in cell capacity of approximately twenty per cent. This comprises an extension of activated life from seventy-two hours to approximately one month, with the addition of five cycles of twenty-five per cent depth. This has been accomplished with no degradation in discharge voltage characteristics.

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III. FACTUAL DATA AND DISCUSSION

A. Separator Evaluation

1. General

Emphasis on evaluation of and improvements upon separator systems has been industry wide in recent years. As a result, studies involving optimization of separator systems for specific applications as well as basic separator materials have been common, in some cases involving duplication of efforts.

During the extended contract period, no fundamental separator studies were carried out. A number of materials had been evaluated on a comparative basis during the first contract year. This phase of study, previously reported in detail (1), included measurements of electrical resistance, speed of wetting and comparative absorbencies and resulted in a separator system of improved capacity retention characteristics.

There is no lack of testing procedures or evaluation methods. Cooper and Fleischer (2) have edited a collection of such screening procedures. Some of the more recent studies have extended the mathematical approach to include tortuosity calculations for membranous materials. This refinement abandons the oversimplification of parallel cylindrical pores and may serve to explain why the resistance of a multilayer membranous system does not appear to be a strictly linear function of the number of layers. The resistance of two layers of cellophane, for instance, may be affected by a mismatch of surface pore openings. In actual cell construction, the effect might be heightened if cell group compression is such that membrane swelling is limited.

There is increased agreement (3,4) that separators retard silver migration by reacting with soluble species of silver, implying a "built-in"

failure mode of separator degradation. Effects of zinc deposition and/or dendritic growths has also been studied in relation to penetration of membranous systems. (5,6)

There remain a number of variables in cell design, however, which cause a need for optimization of separator systems for the various cell designs. The fact that the electrolyte participates in the cell reaction, causing variance in "free" electrolyte during cycle places certain burdens on the separator absorbency. It is desirable for all electrolyte to be retained within the cell element, yet the absorbency of the separator system should be such that electrolyte is available to the active materials. Also, cells of various rate capabilities constructed in a common case dimension frequently employ different plate thickness. This implies varying thickness values for "wicking" absorbent separators if similar wetting characteristics are to be obtained.

It is concluded that while a number of screening procedures may serve as useful criteria for rejection, the ultimate test should include construction of test cells.

2. Preliminary Design Study

a. Object

A few new separator combinations were employed in test cells as described by Table No. I. These cells were intended to evaluate non-woven absorbent materials not previously studied under this contract.

b. Procedure

Test cells were constructed with two thicknesses of 300 cellophane between the positive and negative plates except for Cell No. 2, which contained two wraps of low resistance irradiated teflon. All cells contained

TABLE NO. I

Separator Evaluation - Preliminary Design Study

CELL TYPE	1 & 5	2	3 & 4	6 & 7	8 & 9
Positive "open" separator	#9526 Nylon	#9526 Nylon	EM-470	EM-312	M-1406
Negative "open" separator	R-35-D	R-35-D	EM-470	EM-312	M-1406
Cycle No. 1 Discharge capacity to 1.10v (ampere hours)	10.5	11.1	8.5	10.1	10.75
Cycle No. 5 Discharge capacity to 1.10v	11.9	*	11.8	11.5	12.0

Material Identification

#9526 Nylon - Woven Polyamide - 0.0035 inches thick
EM-312 - "Entangled" nylon fibers with Dynel binder, - 0.0035 inches thick
M-1406 - "Entangled" nylon fibers with Dynel binder, - 0.0025 inches thick
R-35-D - Non-woven rayon fibers, 0.0035 inches thick
EM-470 - Non-woven Dynel

*This cell failed to accept recharge following the initial discharge.

approximately thirty-eight grams each silver and zinc. Cells were activated with 1.400 specific gravity potassium hydroxide solution and allowed to soak for four hours, then discharged at a thirty ampere rate to 1.10 volts per cell. This discharge was conducted to determine the effects of the various separators upon initial performance characteristics. Although only voltage above 1.30 V/cell is considered useful in these studies, discharges were carried further to determine the nature of the "knee" of the various discharge curves.

Cells were then recharged at 2.0 amperes to an end voltage slightly lower than 2.0 V/cell - the point at which there was visible evidence of deposition zinc from the electrolyte. Test cells were then entered into a charged stand at a temperature of $90 \pm 2^\circ\text{F}$. Three discharges of six minute duration at a rate of thirty amperes were conducted in the following month, after which, each of the cells were recharged as described above. At the conclusion of this thirty day period, a full discharge was again conducted.

3. Results

One cell, No. 2, which contained a membrane of irradiated teflon, failed to accept a charge following the initial discharge. Upon examination, it was found that both silver and zinc had penetrated the separator system, resulting in internal shorting. This apparently indicates that this membrane is microporous rather than semipermeable and not well suited to the silver zinc system. During the final discharge, considerable heating of the cells was noted, as indicated by the deformation of some of the cell cases. In battery construction, this effect might be minimized by using copper shim stock to conduct heat away from the cells. The capacity of

all cells was greater following the thirty day period. This is true because cells were charged to a higher state-of-charge than existed at the time of cell construction. This indicates the possible advantages of a "boost" charge immediately following activation and preceding the initial discharge. It is also possible that degradation of the cellulosic membrane lowered its electrical resistance, resulting in sustained voltage at a higher level.

Cells were post mortemed and examined for evidence of near failure. It was found that silver had penetrated to the second layer of cellophane, although the layer closest to the positive plate contained 4.25 times as much silver. Average total silver loss per cell was 1.64 grams, or about 5% of the total. The internal appearance of the cells indicated that these cells were near the end of their useful active life.

B. Negative Material Studies

1. Negative Material Density

a. Object

Material efficiency is a recognized function of porosity or "apparent density". This study was carried out to obtain efficiency data over a range of discharge current densities. These data are useful in determining the ratio of positive and negative materials necessary for optimum performance.

b. Procedure

Zinc plates $1 \frac{5}{8}$ " x 2", the size employed in cells for cycle test, were used to construct negative limited test cells. All negative plates contained 1.45 grams zinc per square inch, but were carefully pressed to varying thicknesses to obtain the desired material densities. These plates were discharged against excess positive material at varying current densities and the resulting efficiencies calculated.

c. Results and Conclusion

Figure No. 1 summarizes test results. Table No. II lists calculations for a specific current density, corresponding to thirty amperes on a cell of the configuration used throughout this investigation. It is apparent that for plates of the weight investigated, efficiency increases with decreasing material density. When converted to a volume basis, as is in the final column of Table No. II, it is apparent that decreased quantities of negative material may result in greater volume efficiency. This effect is most notable at apparent densities in excess of 39 gm/in^3 , as indicated by the final column of the table. It is indicated that for the rate referred to above, an apparent density spread of 40 - 44 gm/in^3 would

result in relatively good capacity control. Lower densities are prone to sloughing of material on cycle or prolonged stand.

TABLE NO. II

Effects of Apparent Density of Zinc
Upon Discharge Efficiency
(At rate of 0.58 amp/in²)

Apparent Density (Gm/in ³)	Thickness (inches)	Efficiency (Per Cent)	Capacity (A. H.)	Capacity (A.H./milli-inch Thickness)
35	0.042	51	1.98	0.047
39	0.038	45	1.75	0.046
44	0.034	38	1.47	0.043
50	0.030	30	1.16	0.039
58	0.026	21	0.81	0.031

2. Zinc Plate Formation Investigation

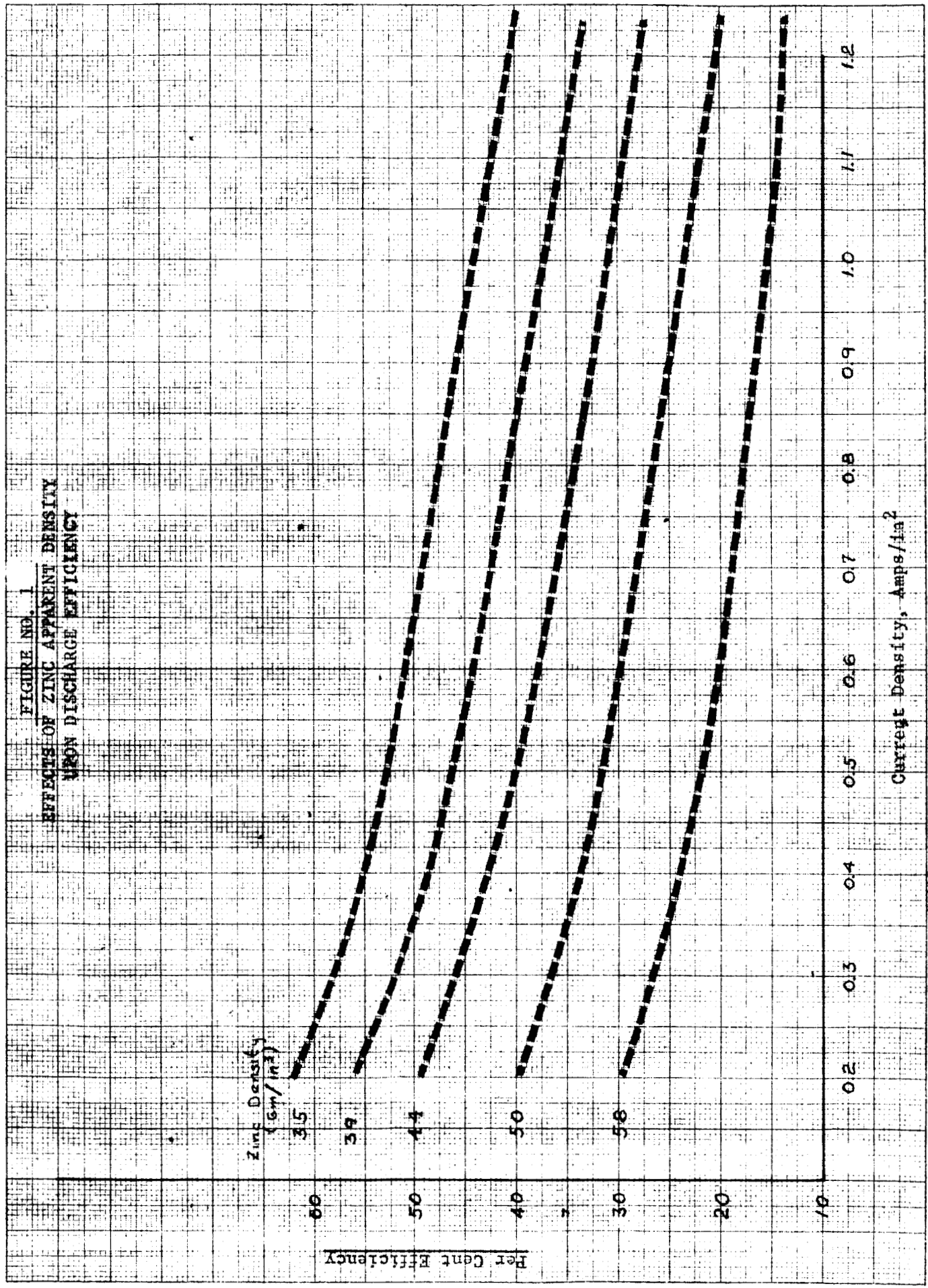
a. Object

This study was of preliminary nature and was intended to reveal whether any of several possible production variables might justify further examination.

b. Procedure

Variations of Eagle-Picher proprietary methods were employed. This procedure consists basically of electrodepositing zinc from a caustic mixture upon the supporting grid. To demonstrate the effects of plating current density, temperature, and plating interval duration, a factorial experiment was initiated involving 40 trials. This trial plan conformed to a balanced plan established by Dr. Robert Hultquist, Oklahoma State

FIGURE NO. 1
 EFFECTS OF ZINC APPARENT DENSITY
 UPON DISCHARGE EFFICIENCY



University, Consultant to Eagle-Picher on Statistics. The factors and levels were as follows:

<u>Factor</u>	<u>Levels</u>				
	0	1	2	3	4
Current Density (amp/in ²)	0.325	0.65	---	---	---
Temperature (°F)	95	110	125	140	---
Plating Interval (Minutes)	15	30	45	60	90

Only two levels of current density were investigated because of equipment limitations. As in previous experiments, levels of factors were equally spaced whenever practical. The factorial series is defined specifically by Table No. III.

Zinc was deposited upon expanded silver grid with interwoven silver wire serving as a terminal lug. Current was impressed for the time interval indicated by Table No. III. The bath temperature was maintained by inserting the plating bath into a small laboratory oven whose temperature was thermostatically controlled. Upon completion of the specified time interval, the plate was removed from the plating solution, washed in flowing water for ten minutes, pressed at 5000 pounds pressure on a laboratory Carver press, then weighed. Plates were then replaced in the plating bath and additional metal was deposited. This procedure was followed for all plates until the total required time interval had elapsed - 180 minutes for a current density of 0.325 amp/in² or 90 minutes at 0.65 amps/in².

Particular emphasis was placed upon the removal of "loose" particles - those not physically bonded to the plate - during the rinse procedure.

As an example, in the case of Trial No. 4 (plate Z-48) a plating current density of 0.325 amps/in² at a bath temperature of 95°F

TABLE NO. XXI

ZINC PLATE FORMATION

Trial	Plate No.	Rate Amp/in	Temp. (°F)	Time Min.	Pass #1 Wt. (gm)	Rate for Pass #1*	Overall Rate
1	Z-31	0.325	95	15	2.84	2.84	0.056
2	Z-32	0.325	95	30	4.52	2.26	0.055
3	Z-47	0.325	95	45	4.63	1.54	0.046
4	Z-48	0.325	95	60	5.55	1.39	0.053
5	Z-49	0.325	95	90	7.36	1.22	0.044
6	Z-33	0.325	110	15	2.69	2.69	0.059
7	Z-7	0.325	110	30	4.63	2.32	0.064
8	Z-34	0.325	110	45	4.62	1.54	0.058
9	Z-8	0.325	110	60	5.59	1.40	0.079
10	Z-9	0.325	110	90	5.70	0.95	0.069
11	Z-35	0.325	125	15	2.64	2.64	0.056
12	Z-36	0.325	125	30	3.90	1.95	0.056
13	Z-50	0.325	125	45	4.70	1.57	0.054
14	Z-23	0.325	125	40	5.58	1.39	0.053
15	Z-24	0.325	125	90	7.33	1.22	0.054
16	Z-37	0.325	140	15	2.55	2.55	0.054
17	Z-16	0.325	140	30	3.65	1.83	0.051
18	Z-38	0.325	140	45	4.65	1.55	0.055
19	Z-17	0.325	140	60	5.99	1.50	0.052
20	Z-18	0.325	140	90	7.04	1.17	0.060
21	Z-1	0.65	95	15	2.75	2.75	0.094
22	Z-2	0.65	95	30	6.92	3.46	0.115
23	Z-3	0.65	95	45	10.12	3.37	0.088
24	Z-39	0.65	95	60	8.00	2.00	0.100
25	Z-40	0.65	95	90	11.53	1.92	0.099
26	Z-4	0.65	110	15	3.45	3.45	0.086
27	Z-5	0.65	110	30	7.35	3.68	0.128
28	Z-6	0.65	110	45	7.32	2.44	0.083
29	Z-41	0.65	110	60	7.57	1.89	0.103
30	Z-42	0.65	110	90	11.72	1.95	0.103
31	Z-19	0.65	110	15	3.51	3.51	0.105
32	Z-20	0.65	125	30	5.45	2.73	0.112
33	Z-21	0.65	125	45	7.08	2.36	0.104
34	Z-43	0.65	125	60	8.47	2.12	0.103
35	Z-44	0.65	125	90	11.33	1.89	0.102
36	Z-13	0.65	140	15	3.43	3.43	0.102
37	Z-14	0.65	140	30	5.45	2.73	0.102
38	Z-15	0.65	140	45	6.99	2.23	0.103
39	Z-45	0.65	140	60	8.35	2.09	0.105
40	Z-46	0.65	140	90	12.16	2.03	0.108

*Rate for Pass No. 1 = (gm Zn deposited during Pass No. 1/Min. for Pass No. 1) x 15

was employed, the three plating intervals alternating with the wash and pressing procedure described above. Responses are also listed in Table No. III and include overall rate of deposit, weight gain during the first plating interval and a weighted rate for the first plating interval referred to the first plating interval as a base. The overall rate was calculated by dividing the "total dry weight of zinc deposited" by the total time. The "pass #1 weight" was the weight of zinc deposited in the first time interval. The "rate of pass #1" is a weighted value compared to the minimum time interval (15 minutes) for each group of plates. For example for Trial #1, (Plate Z-31) the weight of zinc deposited was 2.84 grams for the 15 minute interval; therefore, for the fifteen minute interval $2.84 \text{ grams} / 1 = 2.84 \text{ gm} / 15 \text{ minutes}$. For Trial No. 5 (Plate Z-49) the weight of zinc deposited was 7.36 grams during a 90 minute interval. When weighted (divided by six) this yields a response of 1.22 grams per fifteen minutes. This treatment corrected data for the fact that the current density and time factors were not independent.

Data were analyzed by computer at Oklahoma State University utilizing the following as responses:

- a) The Pass #1 weights,
- b) weighed rate of deposit and
- c) overall rate of zinc deposit

c. Analysis and Discussion

Table Nos. IV - VII display the data as arranged for analysis of main effects with respect to the three responses. In addition, Table Nos. VIII - X data are arranged for testing for interactions of "Temperature x Current Density", "Current Density x Time", and "Temperature x Time", respectively. Each analysis required the calculation indicated on the appropriate table.

A summary of the Analysis of Variance appears in Table No. XI. The "Temperature x Current Density x Time" interaction was taken as a measure of experimental error.

Based upon the rate of zinc deposited, it appears that the preferred conditions include:

- a) Current density of 0.65 amp/in²
- b) 110°F bath temperature
- c) Thirty minute plating interval

Data in Table No. XII compare Zn and ZnO contents of plates washed in flowing water (Plates Z-2, 5, 9, 11, 14, 17, 20, 26 and 29). With those washed in a strong water spray (Plates SZ-4, 8, 18, 20, 22, 2R, 16R and 20R). The oxide content was found to vary from 9.15 to 18.8% in the former group and 8.49 to 11.06% in the latter group. Further, the standard deviation of percentage oxide content in the former group was 3.40 for a flowing water rinse compared to 0.26 for strong spray rinse.

TABLE NO. IV

ZINC PLATE FORMATION

CHEMICAL ANALYSIS

Calculation of Standard Deviation

Trial	Per Cent Zinc	Per Cent ZnO	Trial	Per Cent Zinc	Per Cent ZnO
Z-2	84.50	15.50	SZ-4	88.94	11.06
Z-5	81.20	18.80	SZ-8	91.05	8.95
Z-9	87.60	12.40	SZ-18	90.19	9.81
Z-11	87.80	12.30	SZ-20	89.81	10.19
Z-14	90.85	9.15	SZ-22	91.51	8.49
Z-17	84.30	15.70	SZ-2R	90.71	9.29
Z-20	90.36	9.64	SZ-16R	89.75	10.25
Z-26	86.38	13.72	SZ-20R	89.11	10.89
Z-29	85.62	14.38			
\bar{X}	86.50	13.51	90	90.13	9.87
S*	3.86	3.40		0.288	0.262

* S = Standard Deviation

TABLE NO. V

ANALYSIS OF VARIANCE
 ARRANGED BY TEMPERATURE
 RESPONSE = gm Zn METAL

Trial	80°F	110°F	125°F	140°F	Total
1	2.84	2.69	2.64	2.55	10.72
2	4.52	4.63	3.90	3.65	16.70
3	4.63	4.62	4.70	4.65	18.60
4	5.55	5.59	5.58	5.99	22.71
5	7.36	5.70	7.33	7.04	27.43
6	2.75	3.45	3.51	3.43	13.14
7	6.92	7.35	5.45	5.45	25.17
8	10.12	7.32	7.08	6.99	31.51
9	8.00	7.57	8.47	8.35	32.39
10	11.53	11.72	11.33	12.16	46.74
$\Sigma \bar{x}$	64.22	60.64	59.99	60.26	245.11
\bar{x}	6.422	6.064	5.999	6.026	
Σx^2	489.71	427.93	421.39	434.80	

$$\begin{aligned}\text{Correction Factor} &= (245.11)^2/40 \\ &= 1,501.97\end{aligned}$$

$$\begin{aligned}\text{Total Sum of Squares} &= (\text{Total, each response})^2 - \text{C.F.} \\ &= 271.86\end{aligned}$$

$$\begin{aligned}\text{Temperature S of S} &= ((64.22)^2 + \dots + (60.26)^2/10 - \text{C.F.}) \\ &= 1.18\end{aligned}$$

TABLE NO. VI

ANALYSIS OF VARIANCE

ARRANGED BY CURRENT DENSITY

Trial	0.325 amp/in ²	0.65 amp/in ²	Total
1	2.84	2.75	5.59
2	4.52	6.92	11.44
3	4.63	10.12	14.75
4	5.55	8.00	13.55
5	7.36	11.53	18.89
6	2.69	3.45	6.14
7	4.63	7.35	11.98
8	4.62	7.32	11.94
9	5.59	7.57	13.16
10	5.70	11.72	17.42
11	2.64	3.51	6.15
12	3.90	5.45	9.35
13	4.70	7.08	11.78
14	5.58	8.47	14.05
15	7.33	11.33	18.66
16	2.55	3.43	5.98
17	3.65	5.45	9.10
18	4.65	6.99	11.64
19	5.99	8.35	14.34
20	7.04	12.16	19.20
Σx	96.16	148.95	245.11
\bar{x}	4.81	7.45	
Σx^2	501.9066	1269.1493	3357.9179

Correction Factor = 1,501.97

Total Sum of Squares = 271.86

Current Density S of S = 69.67

TABLE NO. VII

ANALYSIS OF VARIANCE

ARRANGED BY TIME (MINUTES)

Trial	15 Min.	30 Min.	45 Min.	60 Min.	90 Min.	Total
1	2.84	4.52	4.63	5.55	7.36	24.90
2	2.69	4.63	4.62	5.59	5.70	23.23
3	2.64	3.90	4.70	5.58	7.33	24.15
4	2.55	3.65	4.65	5.99	7.04	23.88
5	2.75	6.92	10.12	8.00	11.53	39.32
6	3.45	7.35	7.32	7.57	11.72	37.41
7	3.51	5.45	7.08	8.47	11.33	35.84
8	3.43	5.45	6.99	8.35	12.16	36.38
Σx	23.86	41.87	50.11	55.10	74.17	245.11
\bar{x}	2.98	5.23	6.26	6.89	9.27	
Σx^2	72.324	231.7137	341.4771	291.3854	736.4839	7866.7003

Correction Factor = 1501.97

Total Sum of Squares = 271.86

Time S of S = $((23.86)^2 + \dots + (74.17)^2)/8 - C. F.$
 = 169.35

TABLE NO. VIII

ANALYSIS OF VARIANCE

TEMPERATURE VERSUS CURRENT DENSITY

	<u>0.325 amp/in²</u>	<u>0.65 amp/in²</u>	<u>Total</u>
80°F	2.84	2.75	
	4.52	6.92	
	4.63	10.12	
	5.55	8.00	
	7.36	11.53	
Total	24.90	39.32	64.22
110°F	2.69	3.45	
	4.63	7.35	
	4.62	7.32	
	5.59	7.57	
	5.70	11.72	
Total	23.23	37.41	60.64
125°F	2.64	3.51	
	3.90	5.45	
	4.70	7.08	
	5.58	8.47	
	7.33	11.33	
Total	24.15	35.84	59.99
140°F	2.55	3.43	
	3.65	5.45	
	4.65	6.99	
	5.99	8.35	
	7.04	12.16	
Total	23.88	36.38	60.26
Σx	96.16	148.95	245.11
\bar{x}	24.04	74.48	
Σx_i^2	2313.1198	5553.5805	15,031.4857

$$\begin{aligned} \text{Temperature } \times \text{ CD S of S} &= ((24.90)^2 + \dots + (36.38)^2)/5 - \text{CF} - \text{CD SS} - \\ &\text{Temperature SS} \\ &= 0.52 \end{aligned}$$

TABLE NO. IX

ANALYSIS OF VARIANCE

CURRENT DENSITY VERSUS TIME

0.325 amp/in ²	15 Min.	30 Min.	45 Min.	60 Min.	90 Min.	Total
	2.34	4.52	4.63	5.55	7.36	
	2.69	4.63	4.62	5.59	5.70	
	2.64	3.90	4.70	5.58	7.33	
	2.55	3.65	4.65	5.99	7.04	
Total	10.72	16.70	18.60	22.71	27.43	96.16
0.65 amp/in ²	2.75	6.92	10.12	8.00	11.53	
	3.45	7.35	7.32	7.57	11.72	
	3.51	5.45	7.08	8.47	11.33	
	3.43	5.45	6.99	8.35	12.16	
Total	13.14	25.17	31.51	32.39	46.74	148.95
Σx_i	23.86	41.87	50.11	55.10	74.17	245.11
\bar{x}	11.93	20.94	25.06	27.55	37.09	
Σx_i^2	287.578	912.4189	1338.8401	1564.8462	2937.0325	31,432.85

$$\begin{aligned} \text{CD} \times \text{Time SS} &= ((10.72)^2 + \dots + (46.74)^2)/4 - \text{CF} - \text{CD SS} - \text{Time SS} \\ &= 19.19 \end{aligned}$$

TABLE NO. X

ANALYSIS OF VARIANCE
TEMPERATURE VERSUS TIME

	15 Min.	30 Min.	45 Min.	60 Min.	90 Min.	Total
80°F	2.84 2.75	4.52 6.92	4.63 10.12	5.55 8.00	7.36 11.53	
Total	5.59	11.44	14.75	13.55	18.89	64.22
110°F	2.69 3.45	4.63 7.35	4.62 7.32	5.59 7.57	5.70 11.72	
Total	6.14	11.98	11.94	13.16	17.42	60.64
125°F	2.64 3.51	3.90 5.45	4.70 7.08	5.58 8.47	7.33 11.33	
Total	6.15	9.35	11.78	14.04	18.66	59.99
140°F	2.55 3.43	3.65 5.45	4.65 6.99	5.99 8.35	7.04 12.16	
Total	5.98	9.10	11.64	14.34	19.20	60.26
Σx_i	23.86	41.87	50.11	55.10	74.17	245.11
\bar{x}	5.97	10.47	12.53	13.78	18.54	
Σx_i^2	142.5306	444.6265	634.3841	759.8262	1377.1241	15,031.486

$$\begin{aligned} \text{Temperature x Time SS} &= ((5.59)^2 + \dots + 19.20)^2 - CF - \text{Time SS} - \text{Temperature SS} \\ &= 6.74 \end{aligned}$$

TABLE NO. XI

SUMMARY - ANALYSIS OF VARIANCE

Source	df	Corrected S of S	Mean Squares	F	Significance
Total	39	271.86	---	---	---
Temperature	3	1.18	0.39	0.90	---
C.D.	1	69.67	69.67	162.02	0.005
Time	4	169.35	42.34	98.47	0.005
Interactions					
Temp. x C.D.	3	0.52	0.17	0.40	---
Temp. x Time	12	6.74	0.56	1.30	0.10
C.D. x Time	4	19.19	4.80	11.16	0.005
*Temp. x C.D. x Time	12	5.21	0.43	---	---

* Utilized as a measure of experimental error.

TABLE NO. XII
ZINC PLATE FORMATION
CHEMICAL ANALYSIS

Calculation of Standard Deviation

Trial	Per Cent Zn	Per Cent ZnO	Trial	Per Cent Zn	Per Cent ZnO
Z-2	84.50	15.50	SZ-4	88.94	11.06
Z-5	81.20	18.80	SZ-8	91.05	8.95
Z-9	87.60	12.40	SZ-18	90.19	9.81
Z-11	87.80	12.30	SZ-20	89.81	10.19
Z-14	90.85	9.15	SZ-22	91.51	8.49
Z-17	84.30	15.70	SZ-2R	90.71	9.29
Z-20	90.36	9.64	SZ-16R	89.75	10.25
Z-26	86.38	13.72	SZ-20R	89.11	10.89
Z-29	85.62	14.38			
\bar{X}	86.50	13.51		90.13	9.87
S*	3.86	3.40		0.288	0.262

* S = Standard Deviation

C. Electroformation of the Silver Positive Plate

1. Object

It is obvious that any procedure which results in increased state-of-charge of the active materials at the time of cell construction represents a potential increase in battery capacity. When cells are constructed with sintered silver plates, it is common for the plates to be electroformed in quantity prior to cell group assembly. A number of experiments were carried out to establish a process resulting in the maximum state-of-charge of the silver positive.

2. General Procedure

The state of oxidation of the production positive plate is commonly measured by the weight increase (O_2) attained during the electroformation process, although Dirkse (7) has stated that under certain conditions of charge, oxygen may be adsorbed by the positive plate. Oxygen adsorbed in this manner is apparently released in the gaseous state during discharge without a corresponding yield of ampere hour capacity. It is believed, however, that under the constant current method of formation, the weight increase during charge is an adequate indication of state-of-charge. This is ordinarily expressed as a per cent increase, with a response of 14.83% corresponding to complete conversion of silver to the divalent state. This arises from the increase in molecular weight according to the sequence: $2 Ag-Ag_2O_2$. All plates used in the various series were sintered silver measuring $2 \frac{7}{8} \times 6$ inches. Expanded silver grid of 4/0 mesh served as the support matrix and two 20 gage silver wires as current leads. Specific weights listed in the appropriate tables refer to the active silver weight, exclusive of the mass of the components listed above.

Test cells consisted of the subject plate, Vexar separator, (1/4" x 1/4" mesh polypropylene) nickel sheet anodes and electrolyte. Cells were mounted in individual plastic cases and were series connected during charge. Charge current was impressed across the test cells for the predetermined period of time, after which the plates were removed, washed until KOH free, dried and accurately weighed.

a. First Factorial Experiment (Fractional)

In order to investigate possible effects of charge current density, temperature, plate weight and silver apparent density upon the electroformation process, a fractional factorial experiment was designed following Addelman's Plan No. 11. ("Orthogonal Main-Effect Plans", Addelman-Kempthorne, Iowa State University) The plan, a 1/3 full factorial, utilized twenty-seven trials.

Table XIII describes the test variables, as well as the specific test combinations for each trial.

Approximately 250 ampere hour charge was impressed upon each plate during the formation process after which the individual responses were determined as described above. The following is a summary of test data based upon comparison of mean responses. Data are displayed to show effects of temperature, silver weight and silver density as collected at the three current densities.

High Current Density (0.088 amp/in²)

Temperature

	80°F	100°F	120°F	Mean
Response	9.51	10.44	9.42	9.79

Silver Weight

	8.5 gm/in ²	9.0 gm/in ²	9.5 gm/in ²	Mean
Response	9.99	9.82	9.60	9.80

TABLE NO. XIII

SILVER PLATE FORMATION

FIRST FACTORIAL EXPERIMENT (FRACTIONAL)

Trial	Temp.	C.D.	Weight	Density	Response
1	80	0.022	8.5	90	13.2
2	100	0.088	9.0	110	8.3
3	120	0.044	9.5	100	9.8
4	80	0.022	9.0	100	11.3
5	100	0.088	9.5	90	13.0
6	120	0.044	8.5	110	8.2
7	80	0.022	9.5	110	7.8
8	100	0.088	8.5	100	10.0
9	120	0.044	9.0	90	11.6
10	100	0.044	8.5	90	10.3
11	120	0.022	9.0	110	12.0
12	80	0.088	9.5	100	9.6
13	100	0.044	9.0	100	10.6
14	120	0.022	9.5	90	9.4
15	80	0.088	8.5	110	7.7
16	100	0.044	9.5	110	7.4
17	120	0.022	8.5	100	6.3
18	80	0.088	9.0	90	11.3
19	120	0.088	8.5	90	12.2
20	80	0.044	9.0	110	9.7
21	100	0.022	9.5	100	10.2
22	120	0.088	9.0	100	9.8
23	80	0.044	9.5	90	13.1
24	100	0.022	8.5	110	8.4
25	120	0.088	9.5	110	6.2
26	80	0.044	8.5	100	11.7
27	100	0.022	9.0	90	10.0

Temperature = Temperature, °F

C.D. = Current Density

Weight = Sintered Silver Weight, gm/in²

Density = Silver Apparent Density, gm/in³

Silver Density

90 gm/in ³	100 gm/in ³	110 gm/in ³	Mean
Response 12.19	9.79	7.46	9.81

Medium Current Density (0.044 amp/in²)

Temperature

80°F	100°F	120°F	Mean
Response 11.47	9.42	9.84	10.24

Silver Weight

8.5 gm/in ²	9.0 gm/in ²	9.5 gm/in ²	Mean
Response 10.02	10.63	10.09	10.24

Silver Density

90 gm/in ³	100 gm/in ³	110 gm/in ³	Mean
Response 11.63	10.66	8.78	10.36

Low Current Density (0.022 amp/in²)

Temperature

80°F	100°F	120°F	Mean
Response 10.77	9.54	9.24	9.85

Silver Weight

8.5 gm/in ²	9.0 gm/in ²	9.5 gm/in ²	Mean
Response 10.34	11.11	9.16	10.20

Silver Density

90 gm/in ³	100 gm/in ³	110 gm/in ³	Mean
Response 10.87	9.27	9.39	9.84

More detailed tables of data relative to various test series appear in the Appendix.

Subjective examination of these mean data indicates that over the range of levels investigated, the only clearly significant variable was that of material density.

In order to confirm this as the significant effect, it was decided to analyze the data by the statistical method of Analysis of Variance. Accordingly, data were arranged for this analysis. This arrangement appears in Table XIV in which the data are arranged for the determination of the effect of temperature; Table No. XV in which the data are arranged for the determination of the effect of current density; Table No. XVI in which the data are arranged for the determination of the effect of silver weight; and Table No. XVII in which the data are arranged for the determination of the effect of silver density. The detailed computation is shown at the bottom of each of the tables mentioned above. Each of these required the calculation of a correction factor, total sum of squares, and a sum of squares for the effect being analyzed.

These values are summarized in Table No. XVIII, a summary of the analysis of variance. The total degrees of freedom are 26, since there were 27 samples (trials). Each effect investigated has two degrees of freedom, since there were three levels of each effect involved. Therefore, the residual (error) degrees of freedom are 18, by difference. The corrected sum of squares for each effect (Table No. XIV through XVII) was divided by the number of degrees of freedom to obtain a "mean squares" value for each. Each mean squares value was then divided by the residual (error) mean squares figure to yield the F test value. These F test values when compared to those appearing in Davies "Design and Analysis of Industrial Experiments" yielded the significance values shown. The only effect shown to be significant by this Analysis of Variance was that of the silver density.

This initial experiment lead directly to three additional factorial experiments in which emphasis was placed upon charge input and silver apparent density. These were conducted sequentially and served to optimize the electroformation process.

TABLE NO. XIV

SILVER PLATE FORMATION STUDY

RESPONSES ARRANGED FOR ANALYSIS OF VARIANCE

Effect of Temperature

	High	Medium	Low	Total
	9.77	8.33	13.19	31.29
	8.17	13.04	11.32	32.53
	11.58	9.97	7.80	29.35
	12.00	10.26	9.58	31.84
	9.44	10.58	7.68	27.70
	6.28	7.44	11.29	25.01
	12.24	10.23	9.73	32.20
	9.84	8.39	13.05	31.28
	6.18	10.00	11.65	27.83
Σx_i	85.50	88.24	95.29	269.03
$(\Sigma x_i)^2$	7310.25	7786.30	9080.18	72,377.14

$$\text{Correction Factor} = (269.03)^2 \div 27 = 2680.63$$

$$\begin{aligned} \text{Total Sum of Squares} &= (\text{Total, each response})^2 - CF \\ &= 2782 - 2680 = 102 \end{aligned}$$

$$\begin{aligned} \text{Temperature SS} &= \frac{(7310.25 + 7786.30 + 9080.18)}{9} - CF \\ &= 2686.30 - 2680.63 \\ &= 5.67 \end{aligned}$$

TABLE NO. XV

SILVER PLATE FORMATION STUDY
RESPONSES ARRANGED FOR ANALYSIS OF VARIANCE

Effect of Current Density

	High	Medium	Low	Total
	8.33	9.77	13.19	31.29
	13.04	8.17	11.32	32.53
	9.97	11.58	7.80	29.35
	9.58	10.26	12.00	31.84
	7.68	10.58	9.44	27.70
	11.29	7.44	6.28	25.01
	12.24	9.73	10.23	32.20
	9.84	13.05	8.39	31.28
	6.18	11.65	10.00	27.83
Σx_i	88.15	92.23	88.65	269.03
$(\Sigma x_i)^2$	7770.42	8506.38	7858.82	72,377.14

$$\text{Correction Factor} = (269.03)^2 \div 27 = 2680.63$$

$$\begin{aligned} \text{Total Sum of Squares} &= (\text{Total, each response})^2 - CF \\ &= 2782 - 2680 = 102 \end{aligned}$$

$$\begin{aligned} \text{Current Density SS} &= \frac{(77.40 + 8506.38 + 7858.82) - CF}{9} \\ &= 268.173 - 2680.63 \\ &= 1.10 \end{aligned}$$

TABLE NO. XVI

SILVER PLATE FORMATION STUDY

RESPONSES ARRANGED FOR ANALYSIS OF VARIANCE

Effect of Silver Weight

	High	Medium	Low	Total
	9.77	8.33	13.19	31.29
	13.04	11.32	8.17	32.53
	7.80	11.58	9.97	29.35
	9.58	12.00	10.26	31.84
	9.44	10.58	7.68	27.70
	7.44	11.29	6.28	25.01
	10.23	9.73	12.24	32.20
	13.05	9.84	8.39	31.28
	6.18	10.00	11.65	27.83
Σx_1	86.53	94.67	87.83	269.03
$(\Sigma x_1)^2$	7487.44	8962.41	7714.11	72,377.14

$$\text{Correction Factor} = (269.03)^2 \div 27 = 2680.63$$

$$\begin{aligned} \text{Total Sum of Squares} &= (\text{Total, each response})^2 - CF \\ &= 2782 - 2680 = 102 \end{aligned}$$

$$\begin{aligned} \text{Silver Wt. SS} &= \frac{(7487.44 + 8962.41 + 7714.11)}{9} - CF \\ &= 2684.99 - 2680.63 \\ &= 4.36 \end{aligned}$$

TABLE NO. xvii

SILVER PLATE FORMATION STUDY

RESPONSES ARRANGED FOR ANALYSIS OF VARIANCE

Effect of Apparent Density

	High	Medium	Low	Total
	8.33	9.77	13.19	31.29
	8.17	11.32	13.04	32.53
	7.80	9.97	11.58	29.35
	12.00	9.58	10.26	31.84
	7.68	10.58	9.44	27.70
	7.44	6.28	11.29	25.01
	9.73	10.23	12.24	32.20
	8.39	9.84	13.05	31.28
	6.18	11.65	10.00	27.83
Σx_1	75.75	89.22	104.09	269.03
$(\Sigma x_1)^2$	5733.52	7960.21	10,834.73	72,377.14

$$\text{Correction Factor} = (269.03)^2 \div 27 = 2680.63$$

$$\begin{aligned} \text{Total Sum of Squares} &= (\text{Total, each response})^2 - CF \\ &= 2782 - 2680 = 102 \end{aligned}$$

$$\begin{aligned} \text{Apparent Density SS} &= \frac{(5733.52 + 7960.21 + 10,834.72)}{9} - CF \\ &= 2725.38 - 2680.63 \\ &= 44.75 \end{aligned}$$

TABLE NO. XVIII

SILVER PLATE FORMATION STUDY
SUMMARY OF ANALYSIS OF VARIANCE

Source	df	Sum of Sqs. (Corrected)	Mean Sqs.	F	Significance*
Total	26	102	---	---	---
Temperature	2	5.67	2.84	1.12	---
Current Density	2	1.10	0.55	0.02	---
Silver Weight	2	4.36	2.18	0.86	---
Silver Density	2	44.75	22.38	8.85	0.005
Residual	18	45.57	2.53	---	---

Note: df = degrees of freedom

F = Variance ratio

* Davies: "Design and Analysis of Industrial Experiments, Table C"

F value must exceed 1.70 for significance of 0.1

F value must exceed 3.69 for significance of 0.005

b. Second Factorial Experiment (Fractional)

The second fractional factorial investigated silver apparent densities ranging from 70 - 95 gm/in³ and charge inputs of from 120% to 323% of that theoretically required for complete conversion of the silver material to the divalent oxide. This decreased range of densities was selected in view of the poor responses obtained from the higher density trials of the previous test series. Increments between factor levels were maintained as nearly equal as possible and are described by Table No. XIX. Again, the response was taken as the per cent weight increase observed during the formation process. As the preliminary study indicated no significant effects attributable to current density over the range investigated, a rate of 0.088 amperes per square inch superficial plate surface area was employed.

Table No. XIX summarizes test plan and response data for this second series. Data were again treated by analysis of variance and comparison of means. In all cases, mean data were closely examined to preclude the erroneous conclusion of highly significant single sets of test conditions in opposition to the data trend. The following is a summary of mean response data.

<u>Density</u>	<u>Mean Response (\bar{x})</u>	<u>Overall Mean ($\bar{\bar{x}}$)</u>
70 gm/in ³	13.28%	11.01%
Formation at a silver density of 70 yielded a gain of 2.27 percentage units over the overall mean.		
75 gm/in ³	12.00%	11.01%
Formation at a silver density of 75 yielded a gain of 0.99 percentage units over the overall mean.		
80 gm/in ³	10.91%	11.01%
Formation at a silver density of 80 yielded a loss of 0.10 percentage units from the overall mean.		
85 gm/in ³	10.62%	11.01%
Loss of 0.39 percentage units from the overall mean.		

TABLE NO. XIX

SILVER PLATE FORMATION STUDY

SECOND FACTORIAL EXPERIMENT

Trial	Silver Density Level	Per Cent Charge Level	Final Ag. Wt.	Initial Ag. Wt.	Weight Gain	% Gain	Mean
1	0	0	145.08	126.86	18.22	14.36	13.28
2	0	2	146.98	129.51	17.47	13.49	
3	0	4	146.26	130.60	15.66	11.99	
4	1	1	149.36	131.82	17.54	13.31	12.00
5	1	3	145.01	128.61	16.40	12.75	
6	1	5	144.24	131.20	13.04	9.94	
7	2	0	137.22	123.23	13.99	11.35	10.91
8	2	2	149.42	133.42	16.00	11.99	
9	2	4	143.88	131.53	12.35	9.39	
10	3	1	147.42	132.46	14.96	11.29	10.62
11	3	3	143.12	130.13	13.83	10.63	
12	3	5	143.56	130.57	12.99	9.95	
13	4	0	139.20	127.65	11.55	9.05	10.00
14	4	2	144.26	130.39	13.87	10.64	
15	4	4	143.61	130.20	13.41	10.30	
16	5	1	138.03	126.86	11.17	8.80	9.25
17	5	3	141.45	129.04	12.41	9.62	
18	5	5	144.31	132.00	12.31	9.33	

Overall Mean - 11.01

Per Cent Gain = (Wt. gain/Initial Wt.) x 100

Silver Density: 0 = 70 gm/in³

1 = 75

2 = 80

3 = 85

4 = 90

5 = 95

Per Cent Charge: 0 = 120

1 = 156

2 = 171

3 = 218

4 = 254

5 = 323

<u>Density</u>	<u>Mean Response (\bar{x})</u>	<u>Overall Mean (\bar{x})</u>
90 gm/in ³	10.00%	11.01%
Loss of 1.01 percentage units from the overall mean.		
95 gm/in ³	9.25%	11.01%
Formation at a silver density of 95 yielded a weight increase of 1.76 percentage units below the overall mean.		

On the basis of simple examination of means, the lowest silver density investigated is to be preferred.

Similarly, comparison of means with respect to of charge input is as follows:

<u>% Charge</u>	<u>Mean Response (\bar{x})</u>	<u>Overall Mean (\bar{x})</u>	<u>($\bar{x} - \bar{x}$)</u>
120%	11.59%	11.01%	+0.58%
156%	11.33%	11.01%	+0.32%
171%	12.04%	11.01%	+1.03%
218%	11.00%	11.01%	-0.01%
254%	10.56%	11.01%	-0.45%
323%	9.74%	11.01%	-1.27%

The above analysis indicated that a charge input of 171% of theoretical produced the greatest percentage weight increase on the average. The single trial producing the greatest percentage weight increase, however, was the combination of 70 gm/in³ density and 120% of theoretical charge input. This was a weight increase of 14.36% which is 3.35 percentage units above the overall mean. Table No. XX summarizes results of the analysis of variance, the pertinent calculations of which appear in the Appendix (Tables No. XXXVI through XXXIX). These calculations confirmed the overriding importance of apparent density and served to further reduce the range of interest.

c. Third Factorial Experiment

A third factorial experiment was designed to further investigate the effects of charge input and apparent density. As indicated by Table XXI,

TABLE NO. XX

SILVER PLATE FORMATION STUDY

SECOND FACTORIAL EXPERIMENT

SUMMARY ANALYSIS OF VARIANCE

Source	df	Corrected S of S	M.S.	F	Significance
Total	17	46.96	----	----	----
Silver Density	5	31.25	6.25	7.27	0.005
Per Cent Charge	5	9.67	1.93	2.24	0.05
Residual	7	6.04	0.86	---	---

df = degrees of freedom

F = variance ratio

From Davies' "Design and Analysis of Industrial Experiments"

F must exceed 3.20 for 0.005 Significance

F must exceed 2.10 for 0.05 Significance

TABLE NO. XXI

SILVER PLATE FORMATION STUDY

THIRD FACTORIAL EXPERIMENT

RESPONSES - PER CENT WEIGHT GAIN

Trial	Silver Density	Per Cent Charge	Initial Silver Weight	Final Silver Weight	Per Cent Gain
L-1	71	120	116.76	131.90	12.97
2	70	140	115.15	131.44	14.15
3	70	160	116.83	131.18	12.28
4	76	120	121.25	137.10	13.07
5	75	140	117.95	134.50	14.03
6	75	160	115.55	130.90	13.28
7	80	120	115.26	129.41	12.28
8	79	140	117.91	132.45	12.33
9	80	160	128.24	144.24	12.48
H-1	68	120	125.30	142.09	13.40
2	68	140	124.60	142.75	14.57
3	68	160	125.64	142.93	13.76
4	75	120	126.52	141.90	12.16
5	74	140	127.22	145.01	13.98
6	75	160	127.12	141.96	11.67
7	82	120	124.42	137.73	10.70
8	79	140	126.69	142.58	12.54
9	80	160	130.14	145.52	11.82

Note: The original plan called for densities of 70, 75, and 80 gm/in³. Certain small individual deviations from this plan occurred as indicated by the appropriate column above.

which also lists responses, each of nine possible combinations were duplicated, giving a total of eighteen trials. Tables XLII - XLV (see appendix) list calculations used in evaluating the data by means of analysis of variance, including the estimation of the significance of the variables, density and charge input. As indicated by Table No. XXII, the effects of both density and charge input upon the variability of the process were pronounced, even over the reduced spread of variable levels under investigation. Table No. XLII reveals that the mean response of all plates having densities of 70 gm/in³ was 13.52%, as compared to 11.67 and 12.03 for densities of 75 and 80 gm/in³, respectively. Table No. XLIII indicates that those plates receiving 140% of the charge theoretically required for the conversion of all silver to the divalent state achieved the greatest average weight gain, 13.60%, as compared to 12.43 and 12.54 for those receiving 120% and 160% charge inputs, respectively.

Inspection of these data indicates, therefore, that the greatest oxygen weight gain would be obtained under the following conditions: 1) silver apparent density no greater than 75 gm/in³ and probably nearer 70 gm/in³; and 2) a charge input of approximately 140% of that theoretically required for complete conversion to the divalent state.

d. Interrupted Charge Investigation

1) Procedure

Constant current charging procedures had been confirmed as being ineffective in charging dense positive plates. It was decided to investigate the effects of interrupting the charge process. Data relative to a preliminary experiment are contained in Table No. XXIII. During the "soak", the plates were allowed to remain in the electrolyte, during which time local action processes might occur within the plate structure. This initial experiment provided some indication of increased state-of-charge resulting from the soak periods.

TABLE NO. XXII

SILVER PLATE FORMATION STUDY

THIRD FACTORIAL EXPERIMENT

SUMMARY ANALYSIS OF VARIANCE

Source	df	Corrected Sum of Squares	Mean Square	F	*Significance
Total	17	17.53	----	----	----
Silver Density	2	7.10	3.55	8.45	0.005
Per Cent Charge	2	4.97	2.49	5.93	0.005
Residual	13	5.46	0.42	----	-----

df = Degrees of freedom; F = Variance ratio

*Davies' "Design and Analysis of Industrial Experiments"

F value must exceed 3.20 for 0.005 significance.

TABLE NO. **XXIII**

SILVER PLATE FORMATION EXPERIMENT (FOURTH)

Interrupted Charge Investigation

Responses - Per Cent Weight Gain

Trial	Level of Silver Density	Gm/In ³	Level of Soak Time	Hours	Per Cent Gain	\bar{X}
S-1	0	73	0	4	13.50	14.06
S-2	0	73	1	8	14.20	
S-3	0	73	2	12	14.49	
S-4	1	78	0	4	12.16	12.70
S-5	1	78	1	8	13.35	
S-6	1	78	2	12	12.59	
S-7	2	83	0	4	11.97	12.84
S-8	2	83	1	8	13.26	
S-9	2	83	2	12	13.29	
S-10	3	88	0	4	11.70	12.05
S-11	3	88	1	8	12.36	
S-12	3	88	2	12	12.08	
S-13	4	93	0	4	10.03	10.87
S-14	4	93	1	8	12.32	
S-15	4	93	2	12	10.31	
S-16	5	98	0	4	10.81	10.41
S-17	5	98	1	8	10.17	
S-18	5	98	2	12	10.24	

Experimental Procedure:

- Charge input of 140% that theoretically required for conversion to the divalent state was impressed into the sample plates.
- An open circuit "soak" of the specified duration was allowed.
- An additional 30% charge was introduced into each plate.
- Plates were washed, dried and the response calculated.

A second "soak" investigation was therefore carried out, involving two "soak" periods for each plate as well as increased charge input. Table No. XXIV contains both the plan and response data. Soak times of equal predetermined durations occurred following 140% and 170% of the charge input theoretically required for complete conversion of all silver to the divalent state.

2) Results

This series was the first in which the variation in density, though limited, did not appear to be of overriding importance in the determination of state-of-charge attained. It was also the first series in which an "oxygen pickup" or weight increase of more than 14% was observed with plates of 80 gm/in³ density. This indicates a possible improvement in plate state-of-charge and cell volume efficiency.

TABLE NO. XXIV

SILVER PLATE FORMATION STUDY

FIFTH FACTORIAL EXPERIMENT

INTERRUPTED CHARGE INVESTIGATION

Trial	Silver Density		Soak Time		Weight Gain	% Gain
	Level	gm/in ³	Level	Hours*		
5-52	0	70	0	2 x 3	10.43 gm	14.42
53	0	70	1	2 x 4	10.13	14.03
54	0	70	2	2 x 5	10.21	14.30
55	1	75	0	2 x 3	10.02	14.02
56	1	75	1	2 x 4	9.73	13.65
57	1	75	2	2 x 5	10.31	14.62
58	2	80	0	2 x 3	9.62	13.77
59	2	80	1	2 x 4	9.81	14.13
60	2	80	2	2 x 5	10.09	14.58

*For example, 2 x 3 indicates two three-hour interruptions in the charge procedure.

D. Full Cell Studies

1. Ratio of Materials

a. Purpose

This study was investigated to determine the optimum positive-to-negative material ratio for the cycle method being used.

b. Procedure

Cells were constructed with seven positive plates and eight negative plates, each 1 5/8" x 2". Three ratios of silver to zinc were employed. These were 0.945:1, 1.01:1 and 1.10:1 for Series III, Series IV and Series V, respectively. These ratios represent the total silver and zinc in the cells, whereas the ratios presented in earlier reports reflected gm/in² of these materials.

c. Results

The following table summarizes the results of this study.

TABLE NO. XXV

RATIO OF MATERIALS STUDY

<u>Series</u>	<u>Ratio Ag Zn</u>	<u>C_i (A. H.)</u>	<u>C_f (A. H.)</u>
III	0.945:1	13.55	14.1
IV	1.01:1	14.5	13.9
V	1.10:1	14.65	12.7

NOTE: C_i indicates initial mean capacity

C_f indicates mean capacity following thirty days of cycle testing. It is evident that the initial capacity was greater for those cells having the largest amount of silver, while after thirty days activated life, with the cycle history listed by Table Nos. XXVI-XXVIII, those cells having the

TABLE NO.
RATIO OF MATERIAL
GROUP III

		C E L L								
		1	2	3	4	5	6	7	8	9
CYCLE	1	Vi V max Cap. (AH)	1.37 1.44 12.8	1.39 1.45 13.5	1.35 1.43 13.8	1.36 1.45 13.8	1.36 1.45 13.5	1.36 1.46 14.3	1.36 1.46 14.0	1.38 1.44 13.0
	2	Vi V max	1.39 1.41	1.40 1.42	1.40 1.43	1.41 1.43	1.33 1.38	1.41 1.43	1.40 1.43	1.40 1.43
	3	Vi V max	1.38 1.43	1.37 1.43	1.37 1.43	1.37 1.43	1.36 1.43	1.37 1.43	1.38 1.44	1.38 1.44
	4	Vi V max	1.40 1.43	1.39 1.43	1.40 1.43	1.41 1.43	1.41 1.43	1.38 1.42	1.40 1.42	1.39 1.44
	5	Vi V max	1.33 1.38	1.34 1.39	1.34 1.39	1.35 1.40	1.35 1.39	1.33 1.40	1.35 1.40	1.35 1.39
	6	Vi V max	1.36 1.40	1.37 1.41	1.37 1.42	1.37 1.41	1.37 1.41	1.36 1.41	1.37 1.41	1.37 1.41
	7	Vi V max Cap. (AH)	1.40 1.42 14.3	1.39 1.41 13.8	1.42 1.43 14.5	1.41 1.42 14.5	1.42 1.42 14.0	1.39 1.42 14.0	1.40 1.42 13.8	1.43 1.43 14.3

* Indicates discharge rate other than thirty amperes

** Denotes cell failure

Vi indicates discharge voltage after thirty seconds discharge

Vmax indicates maximum voltage attained during discharge

2


 ALS STUDY

M B E R								
10	11	12	13	14	15	16	17*	18*
1.40	1.42	1.40	1.40	1.40	1.39	1.40		
1.45	1.46	1.46	1.46	1.46	1.46	1.45		
11.5	12.5	14.3	14.5	13.8	14.5	13.8		
1.41	1.40	1.40	1.39	1.39	1.38	1.38	1.38	1.30
1.44	1.43	1.43	1.43	1.43	1.44	1.44	1.44	1.40
1.37	1.36	1.36	1.37	1.37	1.34	1.36	1.36	1.32
1.43	1.44	1.43	1.44	1.44	1.42	1.44	1.43	1.43
1.40	1.40	1.38	1.38	1.39	1.38	1.38	1.37	**
1.43	1.43	1.43	1.43	1.44	1.44	1.45	1.43	
1.35	1.35	1.35	1.35	1.35	1.33	1.34	1.34	
1.38	1.40	1.40	1.40	1.40	1.39	1.39	1.39	
1.37	1.35	1.36	1.36	1.36	1.35	1.36	1.35	
1.40	1.41	1.41	1.41	1.41	1.40	1.41	1.39	
1.39	1.40	1.40	1.41	1.40	1.39	1.39	1.40	
1.41	1.43	1.43	1.43	1.42	1.42	1.43	1.42	
12.5	14.5	14.3	14.0	14.5	14.5	14.5	14.0	

TABLE NO.
RATIO OF MATERIAL
GROUP 1

			C E L L N U								
			1	2	3*	4*	5	6	7	8	9
C Y C L E	1	Vi V max C. (AH)	1.40 1.44 13.5	1.37 1.44 14.3			1.34 1.41 14.0	1.36 1.42 14.3	1.32 1.41 15.8	1.35 1.42 15.3	1.36 1.42 15.0
	2	Vi V max	1.39 1.41	1.38 1.41	1.39 1.42	1.40 1.43	1.41 1.43	1.39 1.42	1.37 1.42	1.37 1.41	1.39 1.42
	3	Vi V max	1.37 1.42	1.35 1.41	1.30 1.38	1.35 1.43	1.36 1.43	1.34 1.44	1.31 1.42	1.33 1.43	1.37 1.44
	4	Vi Vmax	1.38 1.42	1.40 1.44	1.39 1.45	1.40 1.45	1.40 1.44	1.38 1.44	1.36 1.43	1.37 1.43	1.39 1.43
	5	Vi Vmax	1.35 1.40	1.33 1.40	1.35 1.41	1.34 1.40	1.34 1.40	1.34 1.41	1.38 1.38	1.32 1.39	1.31 1.39
	6	Vi Vmax	1.39 1.43	1.36 1.41	1.36 1.44	1.39 1.44	1.37 1.43	1.32 1.41	1.34 1.41	1.37 1.43	1.36 1.42
	7	Vi Vmax C. (AH)	1.36 1.40 13.5	1.34 1.40 13.0	1.33 1.40 13.5	1.32 1.40 13.5	1.33 1.39 13.5	1.33 1.39 14.5	1.30 1.40 13.5	1.32 1.40 13.8	1.34 1.40 14.8

* Indicates discharge rate other than thirty amperes.

Vi indicates discharge voltage after thirty seconds discharge

V max indicates maximum voltage attained during discharge

2

XXVII
ALS STUDY
V

M B E R											
10*	11	12	13	14	15	16	17	18	19	20*	21*
	1.35	1.36	1.38	1.38	1.37	1.36	1.35	1.36	1.36		
	1.41	1.41	1.43	1.43	1.43	1.43	1.44	1.43	1.42		
	14.5	13.5	14.5	15.0	15.0	14.8	14.3	14.0	14.8		
	1.37	1.41	1.38	1.39	1.36	1.39	1.38	1.39	1.39	1.38	1.37
	1.42	1.40	1.43	1.42	1.43	1.42	1.43	1.42	1.43	1.43	1.42
	1.35	1.31	1.35	1.37	1.37	1.30	1.37	1.33	1.36	1.35	1.28
	1.43	1.41	1.42	1.44	1.44	1.39	1.43	1.41	1.43	1.42	1.41
	1.39	1.39	1.39	1.37	1.39	1.38	1.37	1.39	1.38	1.38	1.37
	1.44	1.44	1.44	1.44	1.44	1.44	1.44	1.44	1.45	1.44	1.44
	1.31	1.27	1.33	1.32	1.36	1.39	1.35	1.34	1.34	1.36	1.33
	1.39	1.38	1.39	1.38	1.41	1.36	1.40	1.40	1.39	1.40	1.39
	1.37	1.36	1.37	1.38	1.38	1.33	1.37	1.36	1.38	1.39	1.36
	1.42	1.42	1.42	1.42	1.43	1.42	1.42	1.42	1.43	1.44	1.42
	1.34	1.33	1.38	1.37	1.37	1.36	1.37	1.38	1.36	1.37	1.34
	1.40	1.42	1.42	1.42	1.42	1.43	1.43	1.43	1.41	1.41	1.40
	13.8	14.5	13.8	14.0	13.8	13.8	14.0	14.8	14.8	14.8	13.5

TABLE NO. 1
RATIO OF MATERIAL
GROUP NO.

			C E L L N U M						
			1	2	3	4	5	6	7
C Y C L E	1	Vi	1.36	1.32	1.38	1.34	1.33	1.35	1.35
		Vmax	1.42	1.41	1.43	1.42	1.45	1.46	1.45
		C(AH)	12.5	13.5	14.5	12.0	15.5	16.5	15.5
	2	Vi	1.32	1.37	1.41	1.33	1.36	1.37	1.37
		Vmax	1.38	1.44q	1.44	1.38	1.43	1.43	1.43
	3	Vi	1.30	1.34	1.40	1.17	1.36	1.38	1.37
		Vmax	1.40	1.43	1.44	1.29	1.43	1.44	1.43
	4	Vi	1.31	1.34	1.40	1.32	1.35	1.37	1.36
		Vmax	1.39	1.43	1.42	1.42	1.43	1.44	1.43
	5	Vi	1.29	1.33	1.36	1.31	1.33	1.35	1.37
		Vmax	1.37	1.40	1.41	1.46	1.40	1.42	1.43
	6	Vi	1.34	1.38	1.39	1.37	1.37	1.39	1.38
		Vmax	1.41	1.44	1.44	1.42	1.44	1.44	1.45
	7	Vi	1.33	1.36	1.38	1.35	1.35	1.37	1.37
		Vmax	1.38	1.41	1.41	1.38	1.40	1.42	1.42
		C. (AH)	12.3	13.0	13.0	11.0	12.0	12.5	12.8

* Indicates discharge rate other than thirty amperes.

Vi indicates discharge voltage after thirty seconds discharge

V max indicates maximum voltage attained during discharge

2

IVIII
S STUDY
V

B E R								
8	9	10	11	12	13*	14*	15	16
1.32	1.32	1.36	1.35	1.33			1.32	1.32
1.45	1.45	1.45	1.45	1.46			1.40	1.41
15.5	15.8	13.3	14.5	16.0			14.0	13.5
1.36	1.34	1.40	1.38	1.37	1.34	1.36	1.37	1.37
1.43	1.43	1.42	1.43	1.43	1.43	1.44	1.45	1.45
1.31	1.34	1.38	1.37	1.37	1.36	1.36	1.35	1.35
1.41	1.43	1.43	1.44	1.44	1.41	1.42	1.40	1.40
1.33	1.35	1.36	1.36	1.37	1.35	1.36	1.37	1.36
1.44	1.42	1.44	1.43	1.44	1.44	1.44	1.44	1.42
1.33	1.33	1.35	1.36	1.35	1.32	1.33	1.33	1.33
1.41	1.40	1.39	1.40	1.42	1.39	1.39	1.39	1.38
1.35	1.35	1.39	1.36	1.35	1.35	1.38	1.37	1.37
1.44	1.43	1.43	1.43	1.45	1.43	1.45	1.44	1.43
1.34	1.41	1.42	1.37	1.37	1.36	1.39	1.38	1.39
1.40	1.41	1.41	1.41	1.42	1.42	1.42	1.42	1.42
12.0	11.3	12.0	12.5	12.5	14.0	14.5	14.0	14.0

most zinc and least silver displayed greatest capacity. This is an indication that the capacity degeneration of the zinc plate proceeds more rapidly than the loss from the positive group. Therefore, cells tend to be negative-limited following thirty days activated life. Interpolation of these data indicate that a silver zinc ratio 0.97:1.00 might yield, under these specific discharge conditions, a relatively stable capacity over the useful life of the cell.

2. Electrolyte Additive Study

a. Purpose

The objective of this study was to determine whether any of several electrolyte additives might result in significant benefits in capacity retention over a period of thirty days.

b. Procedure

The following solutions of additives in 1.400 specific gravity potassium hydroxide electrolyte were prepared by The Eagle-Picher Analytical Laboratory:

- (a) Kaolin (20% saturated, 80% saturated)
- (b) Polyethylene Oxide (20% saturated, 80% saturated)
- (c) Lithium Hydroxide (20% saturated, 80% saturated)
- (d) Aluminum Hydroxide (20% saturated, 80% saturated)
- (e) Elvanol (PVA), (20% saturated, 80% saturated)
- (f) $\text{Na}_x(\text{SiO}_2)_y$ (1 wt. %, 10 wt. %)

Cells of the same size and nominal capacity as used previously were activated with twenty cc of the appropriate electrolyte. Certain cells were discharged to 1.30 end voltage on the initial cycle to establish full capacity. Others were discharged for 7.5 minutes at 30 amperes, or approximately 25% of full depth. Results of this and other cycles are included in Table No. XXX. Table No. XXIX specifies the electrolyte employed in each cell.

TABLE NO. XXIX
ELECTROLYTE ADDITIVES

CELL NUMBERS	ADDITIVES
1 - 10	None
11 - 12	Polyethylene Oxide (20% sat.)
13 - 14	Polyethylene Oxide (80% sat.)
15 - 16	Kaolin (20% sat.)
17 - 18	Kaolin (80% sat.)
19 - 21	LiOH (20% sat.)
22 - 24	LiOH (80% sat.)
31 - 33	Al ₂ O ₃ (20% sat.)
34 - 36	Al ₂ O ₃ (80% sat.)
37 - 39	Elvanol (20% sat.)
40 - 42	Elvanol (80% sat.)
43 - 45	Na _x (SiO ₂) _y , 1% by wt.
46 - 48	Na _x (SiO ₂) _y , 10% by wt.
49 - 51	None

TABLE NO.
ELECTROLYTE
GROUP NO.

			CELL NUMBER										
			1	2	3	4	5	6	7	8	9	10	11
CYCLE NO.	1	Vi V Max Cap. (AH)	1.34 1.42 12.0	1.30 1.42 13.25	1.31 1.40 10.75	1.33 1.42 12.0	1.32 1.43 13.25	1.32 1.41 11.0	1.34 1.40 9.0*	1.36 1.44 11.5*	1.34 1.39 8.5*	1.32 1.40 10.0*	1.33 1.39
	2	Vi V Max	1.38 1.40	1.38 1.40	1.37 1.40	1.37 1.41	1.36 1.41	1.37 1.40	1.42 1.42	1.39 1.42	1.39 1.42	1.30 1.40	1.37 1.41
	3	Vi V Max	1.41 1.43	1.39 1.41	1.38 1.41	1.40 1.42	1.39 1.42	1.40 1.41	1.41 1.42	1.41 1.43	1.41 1.43	1.37 1.42	1.36 1.40
	4	Vi V Max	1.37 1.39	1.37 1.38	1.36 1.38	1.36 1.39	1.33 1.38	1.34 1.38	1.39 1.40	1.37 1.40	1.37 1.39	1.33 1.38	1.36 1.38
	5	Vi V Max	1.39 1.41	1.39 1.41	1.37 1.40	1.39 1.42	1.38 1.42	1.39 1.41	1.42 1.43	1.42 1.43	1.41 1.42	1.37 1.41	1.43 1.41
	6	Vi V Max	1.37 1.38	1.37 1.38	1.34 1.37	1.37 1.38	1.34 1.38	1.37 1.38	1.38 1.39	1.37 1.40	1.37 1.38	1.31 1.36	1.33 1.37
	7	Vi V Max Cap. (AH)	1.36 1.38 13.0	1.37 1.38 12.5	1.34 1.38 12.5	1.36 1.39 12.5	1.35 1.39 13.0	1.36 1.38 12.5	1.37 1.38 13.0	1.37 1.39 13.5	1.37 1.38 13.0	1.32 1.37 12.5	1.34 1.38 12.0

* Indicates discharge rate other than thirty amperes

** Denotes cell failure

Vi indicates discharge voltage after 30 seconds

V Max indicates maximum voltage attained during discharge

2

~~XXX~~
STUDY
VII

3R

12	13	14	15	16	17	18	19	20	21	22
1.33 1.42	1.33 1.42	1.34 1.42	1.33 1.41	1.35 1.42	1.31 1.40	1.32 1.40	1.34 1.41	1.33 1.40	1.31 1.41	1.33 1.41
1.37 1.42	1.36 1.42	1.38 1.42	1.35 1.42	1.37 1.42	1.33 1.42	1.31 1.42	1.37 1.42	1.36 1.43	1.33 1.41	1.34 1.41
1.37 1.41	1.36 1.41	1.38 1.42	1.34 1.42	1.35 1.42	1.33 1.41	1.33 1.42	1.35 1.42	1.35 1.42	1.34 1.40	1.34 1.41
1.37 1.39	1.36 1.38	1.36 1.40	1.33 1.38	1.34 1.39	1.33 1.38	1.33 1.38	1.35 1.38	1.30 1.37	1.37 1.37	1.33 1.38
1.42 1.41	1.41 1.41	1.41 1.41	1.41 1.42	1.43 1.43	1.40 1.42	1.38 1.41	1.39 1.40	1.38 1.40	1.38 1.39	1.38 1.40
1.35 1.35	1.34 1.37	1.34 1.38	1.34 1.38	1.34 1.39	1.33 1.38	1.32 1.38	1.32 1.37	1.32 1.37	1.34 1.37	1.33 1.39
1.37 1.35 12.25	1.34 1.37 12.25	1.34 1.37 11.5	1.34 1.38 12.0	1.36 1.39 12.5	1.33 1.37 12.0	1.33 1.37 12.25	1.33 1.37 11.5	1.31 1.35 12.5	1.35 1.38 12.0	1.34 **

(continued next page)

TABLE NO. XX
ELECTROLYTE
GROUP NO.

			CELL NUM										
			23	24	31	32	33	34	35	36	37	38	39
CYCLE NO.	1	Vi V Max	1.31 1.39	1.31 1.40	1.35 1.42	1.37 1.43	1.38 1.42	1.35 1.42	1.34 1.42	1.33 1.42	1.35 1.43	1.35 1.42	1.35 1.43
	2	Vi V Max	1.32 1.41	1.32 1.41	1.37 1.41	1.39 1.43	1.37 1.42	1.33 1.42	1.36 1.42	1.31 1.41	1.39 1.42	1.37 1.43	1.39 1.43
	3	Vi V Max	1.33 1.41	1.32 1.40	1.31 1.38	1.36 1.42	1.36 1.42	1.34 1.41	1.35 1.41	1.29 1.39	1.35 1.41	1.37 1.42	1.38 1.42
	4	Vi V Max	1.33 1.38	1.35 1.38	1.33 1.37	1.33 1.39	1.34 1.39	1.33 1.39	1.33 1.38	1.28 1.37	1.33 1.38	1.35 1.39	1.35 1.40
	5	Vi V Max	1.37 1.40	1.37 1.40	1.33 1.40	1.34 1.41	1.33 1.41	1.33 1.41	1.34 1.40	1.35 1.39	1.35 1.43	1.34 1.40	1.36 1.41
	6	Vi V Max	1.33 1.39	1.33 1.39	1.36 1.37	1.35 1.38	1.35 1.37	1.34 1.37	1.35 1.36	1.30 1.36	1.37 1.37	1.35 1.38	1.38 1.38
	7	Vi V Max Cap. (AH)	1.33 **	1.33 **	1.37 1.37 13.0	1.35 1.39 14.5	1.36 1.38 13.5	1.36 1.37 13.5	1.35 1.36 11.0	1.31 1.38 14.5	1.37 1.39 14.0	1.35 1.38 12.0	1.39 1.39 13.0

** Denotes cell failure

Vi indicates discharge voltage after 30 seconds

V Max indicates maximum voltage attained during discharge

2

I (Cont.)

STUDY
VII

BER

	40	41	42	43	44	45	46	47	48	49	50	51
	1.35 1.42	1.38 1.43	1.37 1.43	1.32 1.39	1.34 1.39	1.31 1.40	1.26 1.28	1.25 1.30	1.25 1.30	1.36 1.42	1.37 1.43	1.35 1.42
	1.36 1.43	1.41 1.42	1.40 1.42	1.33 1.41	1.29 1.40	1.28 1.39	1.20 1.30	1.20 1.32	1.17 1.30	1.33 1.42	1.36 1.42	1.35 1.42
	1.36 1.42	1.38 1.42	1.38 1.42	1.33 1.41	1.32 1.40	1.28 1.40	1.22 1.27	1.20 1.28	1.17 1.26	1.34 1.42	1.35 1.42	1.33 1.41
	1.34 1.40	1.37 1.38	1.38 1.38	1.32 1.37	1.30 1.37	1.28 1.37	1.28 1.28	1.27 1.29	1.26 1.29	1.33 1.38	1.34 1.39	1.32 1.37
	1.35 1.39	1.43 1.43	1.42 1.42	1.37 1.40	1.33 1.39	1.32 1.39	1.29 1.28	1.30 1.30	1.28 1.30	1.38 1.40	1.38 1.41	1.37 1.41
	1.33 1.37	1.35 1.37	1.34 1.37	1.32 1.36	1.29 1.36	1.29 1.33	1.28 1.32	1.27 1.27	1.25 1.25	1.32 1.37	1.32 1.38	1.32 1.37
	1.34 1.36 11.0	1.35 1.37 11.5	1.34 1.37 12.0	1.31 1.35 11.5	1.31 1.36 11.5	1.31 1.36 11.5	1.31 **	1.31 **	1.31 **	1.32 1.38 11.5	1.33 1.38 12.5	1.33 1.37 13.0

c. Results and Conclusions

The only obvious significant effects were those caused by deleterious additives. For instance, both concentrations of $\text{Na}_x(\text{SiO}_2)_y$ resulted in poor discharge voltage. The greater concentration of this additive resulted in complete disability of cells to reach acceptable discharge voltage at a 30 ampere rate, following 30 days activated life. Discharge capabilities of cells using the higher concentration of lithium hydroxide similarly failed to discharge satisfactorily, while the lower concentration revealed no apparently significant effect. The mean capacity of all cells except those described above was 12.5 ampere-hours. The mean capacity of cells having no electrolyte additive was also 12.6 ampere-hours. This appears coincidental, as the six cells having aluminum hydroxide additive displayed an average capacity of 13.3 ampere-hours, while the remaining cells with additives (polyethylene oxide, kaolin and elvanol) exhibited an average capacity of 12.10 ampere-hours. It is indicated, therefore, that for this particular regime of testing, the addition of aluminum hydroxide might be beneficial.

3. Effect of Cell Tightness and Quantity of Electrolyte

a. Object

The object of this phase of study is to determine whether certain combinations of cell tightness and quantity of electrolyte result in improvement in voltage regulation or capacity retention.

b. Procedure

Cells used in this series were similar to those described above with respect to active material weights and type of separator system. In all cells, the separator system consisted of non-woven nylon fabric, two wraps of cellophane and nonwoven rayon. Shims of two thicknesses were used to

provide the variable of tightness. Electrolyte volumes of 19, 19.5 and 20 cc's were selected. All possible combinations of these variables were employed as indicated by Table No. XXXI.

c. Results and Conclusions

Table No. XXXII displays cycle data for these cells. Mean responses for the nine types of cells ranged from 11.1 to 12.4 ampere hours, with no definite indication of any significant effect, either beneficial or deleterious. This is taken to indicate that a) the quantity of electrolyte may be reduced to nineteen cc's and b) the cell capacity may be increased by adding more active material in the same volume. It is notable that no cell failures occurred in this test series.

4. Prototype Cell Investigation

a. Cell Design

Development work concluded during the final contract month with the cycle evaluation of cells of improved design. Design changes during the contract period are more fully outlined in the following section, Section IV.

b. Test Procedure

As in previous test series, cells were activated with electrolyte, then entered into stand at +90°F. As indicated by Table No. XXXIV, which summarizes cycle data for this test series, certain cells were discharged fully on the initial cycle, while others were discharged approximately 25% of the mean full capacity. A thirty ampere rate was used for all discharges. Cells were subjected to seven discharges during a thirty day period, at least one of these cycles being carried to an end voltage of 1.3 volts per cell.

TABLE NO. XXXI

EFFECT OF CELL TIGHTNESS AND QUANTITY OF ELECTROLYTE

Cell Numbers	Volume of Electrolyte	Shim Thickness
1 - 7	19.0	-----
8 - 14	19.5	-----
15 - 20	20.0	-----
21 - 27	19.0	0.010"
28 - 34	19.5	0.010"
35 - 40	20.0	0.010"
41 - 47	19.0	0.020"
48 - 54	19.5	0.020"
55 - 60	20.0	0.020"

TABLE NO. 1
EFFECT OF CELL TIGHTNESS AND Q
GROUP NO.

		CELL NUMBER													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Vi	1.36	1.36	1.32	1.37	1.35	1.35	1.37	1.35	1.34	1.35	1.36	1.36	1.36	1.36
	V Max.	1.42	1.42	1.41	1.42	1.42	1.42	1.42	1.42	1.42	1.43	1.41	1.41	1.41	1.42
	Cap.AH	14.5	14.5							15.0	15.75				
2	Vi	1.42	1.41	1.39	1.41	1.40	1.39	1.41	1.40	1.41	1.39	1.40	1.40	1.39	1.39
	V Max.	1.44	1.43	1.40	1.41	1.41	1.41	1.42	1.41	1.42	1.42	1.42	1.40	1.41	1.40
3	Vi	1.38	1.37	1.37	1.40	1.39	1.39	1.40	1.39	1.38	1.36	1.38	1.38	1.38	1.38
	V Max.	1.42	1.42	1.37	1.40	1.39	1.39	1.40	1.39	1.42	1.42	1.40	1.39	1.39	1.39
4	Vi	1.37	1.35	1.36	1.38	1.36	1.36	1.38	1.37	1.36	1.35	1.37	1.38	1.38	1.37
	V Max.	1.41	1.41	1.36	1.38	1.37	1.37	1.38	1.38	1.41	1.41	1.38	1.38	1.38	1.37
5	Vi	1.35	1.36	1.37	1.37	1.37	1.37	1.37	1.37	1.35	1.34	1.37	1.37	1.37	1.35
	V Max.	1.39	1.38	1.38	1.38	1.35	1.38	1.38	1.38	1.39	1.39	1.38	1.37	1.37	1.36
6	Vi	1.37	1.37	1.38	1.38	1.37	1.36	1.38	1.38	1.35	1.35	1.33	1.33	1.34	1.33
	V Max.	1.40	1.40	1.38	1.38	1.38	1.38	1.38	1.38	1.40	1.41	1.38	1.35	1.37	1.34
7	Vi	1.32	1.34	1.34	1.33	1.33	1.32	1.34	1.34	1.33	1.33	1.34	1.33	1.34	1.34
	V Max.	1.38	1.39	1.37	1.37	1.38	1.38	1.38	1.38	1.39	1.39	1.37	1.37	1.38	1.38
	Cap.AH	11.75	10.5	11.5	11.5	12.25	11.5	12.25	11.75	10.5	12.5	11.5	12.5	12.0	12.0

		CELL NUMBER													
		31	32	33	34	35	36	37	38	39	40	41	42	43	44
1	Vi	1.33	1.36	1.36	1.35	1.35	1.35	1.35	1.34	1.33	1.32	1.35	1.32	1.36	1.36
	V Max.	1.41	1.42	1.42	1.42	1.42	1.42	1.42	1.42	1.43	1.43	1.43	1.43	1.41	1.41
										15.0	14.5	14.0	14.5		
2	Vi	1.38	1.39	1.38	1.39	1.39	1.39	1.39	1.38	1.39	1.39	1.42	1.44	1.43	1.40
	V Max.	1.40	1.41	1.41	1.41	1.41	1.41	1.41	1.40	1.43	1.42	1.44	1.44	1.43	1.42
3	Vi	1.38	1.39	1.38	1.39	1.40	1.39	1.39	1.38	1.36	1.37	1.38	1.37	1.35	1.38
	V Max.	1.39	1.39	1.39	1.39	1.40	1.39	1.39	1.38	1.41	1.41	1.42	1.42	1.38	1.39
4	Vi	1.37	1.36	1.37	1.37	1.38	1.37	1.37	1.37	1.34	1.36	1.38	1.36	1.36	1.37
	V Max.	1.37	1.37	1.37	1.37	1.38	1.37	1.38	1.37	1.41	1.41	1.41	1.41	1.37	1.37
5	Vi	1.37	1.37	1.37	1.37	1.38	1.37	1.37	1.37	1.34	1.36	1.38	1.37	1.36	1.35
	V Max.	1.37	1.37	1.37	1.37	1.38	1.37	1.37	1.37	1.41	1.41	1.41	1.41	1.36	1.37
6	Vi	1.34	1.34	1.34	1.33	1.34	1.34	1.35	1.34	1.34	1.35	1.37	1.35	1.34	1.33
	V Max.	1.37	1.35	1.37	1.34	1.35	1.35	1.37	1.35	1.40	1.40	1.39	1.40	1.34	1.36
7	Vi	1.37	1.37	1.36	1.36	1.37	1.36	1.37	1.36	1.35	1.37	1.36	1.36	1.34	1.32
	V Max.	1.38	1.38	1.39	1.39	1.39	1.39	1.39	1.38	1.39	1.40	1.39	1.40	1.38	1.38
	Cap.AH	12.0	11.50	12.0	11.75	12.5	11.5	11.5	12.5	11.5	10.5	11.5	10.0	11.0	12.5

Vi Indicates Discharge Voltage After 30 Seconds.

V Max. Indicates Maximum Voltage Attained During Discharge.

2

R															
15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
1.36	1.37	1.36	1.37	1.34	1.34	1.37	1.34	1.34	1.35	1.35	1.35	1.35	1.36	1.35	1.33
1.42	1.42	1.42	1.42	1.42	1.42	1.43	1.43	1.41	1.41	1.41	1.41	1.42	1.42	1.43	1.42
				15.0	13.75	14.5	15.0							15.0	15.0
1.39	1.39	1.40	1.40	1.40	1.41	1.41	1.39	1.40	1.40	1.39	1.40	1.40	1.41	1.41	1.41
1.41	1.40	1.41	1.42	1.43	1.43	1.44	1.40	1.41	1.41	1.41	1.42	1.42	1.43	1.44	1.43
1.38	1.39	1.37	1.38	1.37	1.39	1.38	1.38	1.37	1.39	1.38	1.38	1.38	1.39	1.38	1.39
1.39	1.39	1.40	1.40	1.41	1.42	1.42	1.42	1.38	1.39	1.39	1.39	1.40	1.39	1.42	1.42
1.36	1.38	1.38	1.39	1.36	1.38	1.36	1.35	1.35	1.34	1.36	1.37	1.36	1.37	1.35	1.36
1.36	1.38	1.39	1.39	1.41	1.42	1.41	1.41	1.36	1.36	1.37	1.37	1.38	1.37	1.41	1.41
1.36	1.37	1.37	1.35	1.35	1.37	1.34	1.34	1.34	1.36	1.36	1.36	1.37	1.37	1.35	1.37
1.36	1.37	1.37	1.37	1.40	1.40	1.39	1.39	1.37	1.36	1.37	1.37	1.37	1.37	1.39	1.40
1.34	1.35	1.34	1.35	1.33	1.38	1.34	1.33	1.34	1.33	1.34	1.34	1.33	1.33	1.33	1.34
1.35	1.36	1.36	1.36	1.39	1.40	1.41	1.39	1.35	1.35	1.36	1.35	1.35	1.35	1.40	1.41
1.34	1.35	1.34	1.34	1.34	1.37	1.33	1.33	1.33	1.33	1.33	1.33	1.35	1.34	1.35	1.35
1.38	1.38	1.39	1.38	1.39	1.39	1.40	1.40	1.40	1.39	1.40	1.39	1.40	1.40	1.41	1.40
11.75	12.0	13.0	12.0	10.5	12.0	11.0	12.0	13.0	12.5	12.5	12.5	13.25	11.25	11.75	11.75

R															
45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
1.35	1.35	1.36	1.36	1.34	1.34	1.36	1.36	1.36	1.37	1.33	1.35	1.35	1.36	1.35	1.36
1.41	1.41	1.42	1.42	1.42	1.42	1.40	1.41	1.42	1.42	1.41	1.42	1.42	1.42	1.43	1.43
				15.25	14.5									15.0	15.25
1.40	1.39	1.40	1.38	1.39	1.39	1.38	1.37	1.38	1.38	1.38	1.37	1.39	1.39	1.41	1.40
1.41	1.40	1.41	1.41	1.43	1.43	1.39	1.40	1.40	1.39	1.40	1.40	1.40	1.41	1.43	1.42
1.38	1.38	1.37	1.37	1.37	1.38	1.39	1.36	1.38	1.36	1.39	1.36	1.39	1.40	1.38	1.37
1.39	1.38	1.38	1.38	1.41	1.41	1.39	1.38	1.38	1.38	1.39	1.38	1.39	1.40	1.41	1.41
1.36	1.37	1.34	1.35	1.35	1.37	1.38	1.37	1.37	1.35	1.35	1.37	1.37	1.39	1.38	1.37
1.37	1.37	1.37	1.37	1.41	1.41	1.38	1.37	1.37	1.36	1.36	1.37	1.37	1.39	1.41	1.41
1.36	1.35	1.35	1.36	1.37	1.38	1.38	1.38	1.37	1.37	1.38	1.38	1.38	1.38	1.38	1.36
1.36	1.35	1.35	1.36	1.41	1.38	1.38	1.38	1.37	1.37	1.38	1.38	1.38	1.38	1.41	1.41
1.33	1.33	1.31	1.33	1.33	1.35	1.34	1.34	1.33	1.33	1.33	1.35	1.34	1.36	1.35	1.37
1.35	1.34	1.34	1.35	1.40	1.40	1.35	1.35	1.34	1.35	1.34	1.35	1.35	1.36	1.40	1.41
1.31	1.35	1.33	1.33	1.33	1.35	1.36	1.35	1.34	1.35	1.34	1.36	1.35	1.37	1.38	1.37
1.38	1.37	1.37	1.37	1.39	1.40	1.37	1.38	1.37	1.38	1.37	1.38	1.38	1.39	1.40	1.40
11.5	11.5	12.0	12.0	10.0	10.5	11.75	11.0	11.5	11.0	11.0	11.0	13.5	12.0	11.0	11.0

c. Cell Capacities

Cell capacity data as a function of cell test conditions are revealed by Table Nos. XXXIII and XXXV.

As a previous study indicated a possible benefit from the addition of Al_2O_3 to the electrolyte, ten of the sixty-five cells of this series (Cell Nos. 25 - 34) employed electrolyte 20% saturated with this additive.

TABLE NO. XXXIII

EFFECT OF Al_2O_3 UPON DISCHARGE CAPACITY

	Initial Capacity	Final Capacity
Al_2O_3	14.4 A.H.	15.6 A.H.
No Al_2O_3	15.7	13.9

Table No. XXXIV reveals that four cells failed during cycle evaluation. All failed cells were disassembled and carefully examined. Of these, Cell No. 13 failed to accept a recharge following the initial 25% depth cycle. This was attributed to penetration of the separator system by a minute piece of grid wire. This is regarded as abnormal failure and does not indicate a design fault. The remaining cells which failed were found to have inadequate plate-to-terminal electrical connection because of weakening of the grid lug structure, apparently the result of excessive heating during an assembly process. There was no evidence of conductive paths of active materials penetrating the separator system in any cell, although both silver and zinc were observed; nor was there any indication of excessive loss of active materials from the plates. It is indicated that the use of Al_2O_3 in the electrolyte tends to stabilize cell capacity on stand. Over

TABLE NO. XXXIV
PROTOTYPE CELL EVALUATION

		CELL NUMBER															
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1	V _i	1.34	1.35	1.38	1.38	1.38	1.34	1.38	1.37	1.39	1.34	1.35	1.33	1.39	1.35	1.38	1.33
	V _m	1.44	1.47	1.47	1.47	1.48	1.43	1.43	1.44	1.43	1.43	1.43	1.44	1.43	1.44	1.44	1.44
	C	15.25	16.25	15.0	14.5	16.25	15.0										
2	V _i	1.39	1.44	1.43	1.42	1.41	1.38	1.40	1.39	1.33	1.38	1.37	1.35	*	1.35	1.41	1.39
	V _m	1.43	1.44	1.43	1.43	1.43	1.43	1.43	1.44	1.42	1.43	1.43	1.43		1.43	1.46	1.43
3	V _i	1.38	1.37	1.42	1.42	1.38	1.37	1.38	1.37	1.34	1.35	1.35	1.33		1.34	1.40	1.32
	V _m	1.41	1.40	1.42	1.42	1.40	1.42	1.42	1.42	1.42	1.42	1.41	1.41		1.41	1.44	1.41
4	V _i	1.40	1.35	1.42	1.42	1.38	1.37	1.39	1.37	1.30	1.38	1.33	1.32		1.35	1.40	1.36
	V _m	1.42	1.42	1.43	1.43	1.41	1.42	1.42	1.44	1.43	1.44	1.43	1.43		1.43	1.45	1.41
5	V _i	1.34	1.33	1.38	1.39	1.35	1.35	1.37	1.34	1.27	1.32	1.31	1.24		1.30	1.38	1.31
	V _m	1.39	1.40	1.41	1.41	1.39	1.41	1.40	1.40	1.40	1.40	1.40	1.40		1.40	1.43	1.40
6	V _i	1.36	1.33	1.38	1.39	1.36	1.35	1.38	1.36	1.28	1.34	1.32	1.33		1.34	1.40	1.34
	V _m	1.40	1.40	1.41	1.42	1.39	1.40	1.41	1.41	1.40	1.40	1.39	1.40		1.40	1.43	1.39
7	V _i	1.32	1.31	1.37	1.37	*	1.33	1.37	1.36	1.31	1.34	1.33	1.32		1.32	1.39	1.31
	V _m	1.40	1.40	1.41	1.41		1.41	1.41	1.41	1.41	1.40	1.40	1.40		1.40	1.43	1.41
	C	15.5	14.0	14.0	13.5		15.5	15.0	16.5	16.0	15.0	15.0	14.5		15.0	13.5	15.5

		CELL NUMBER															
		34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49
1	V _i	1.35	1.38	1.37	1.34	1.34	1.36	1.34	1.36	1.35	1.33	1.37	1.37	1.38	1.34	1.34	1.35
	V _m	1.41	1.46	1.46	1.46	1.46	1.46	1.45	1.46	1.44	1.45	1.45	1.46	1.43	1.45	1.44	1.45
	C	14.0	15.0	16.0	15.5	16.0	15.0	16.0	16.0	15.5	16.0	13.5	14.5	13.5	15.5	16.0	16.0
2	V _i	1.42	1.38	1.37	1.37	1.33	1.34	1.33	1.36	1.33	1.38	1.38	1.37	1.38	1.38	1.38	1.36
	V _m	1.43	1.41	1.40	1.40	1.41	1.40	1.40	1.41	1.41	1.41	1.40	1.40	1.40	1.40	1.40	1.40
3	V _i	1.38	1.38	1.37	1.35	1.36	1.37	1.37	1.37	1.36	1.37	1.40	1.41	1.41	1.38	1.39	1.33
	V _m	1.41	1.42	1.40	1.40	1.40	1.41	1.41	1.41	1.40	1.41	1.41	1.42	1.42	1.41	1.41	1.41
4	V _i	1.39	1.40	1.39	1.37	1.38	1.40	1.40	1.40	1.39	1.39	1.42	1.44	1.44	1.43	1.43	1.35
	V _m	1.42	1.43	1.43	1.40	1.41	1.42	1.43	1.43	1.42	1.40	1.42	1.43	1.43	1.43	1.43	1.40
5	V _i	1.39	1.38	1.36	1.38	1.35	1.38	1.37	1.37	1.36	1.34	1.40	1.41	1.42	1.39	1.35	1.35
	V _m	1.42	1.41	1.39	1.38	1.39	1.41	1.39	1.38	1.38	1.40	1.42	1.43	1.43	1.40	1.40	1.40
6	V _i	1.34	1.37	1.38	1.33	1.37	1.36	1.37	1.37	1.36	1.38	1.39	1.40	1.40	1.40	1.37	1.34
	V _m	1.39	1.42	1.41	1.38	1.40	1.41	1.40	1.41	1.42	1.42	1.42	1.42	1.42	1.42	1.41	1.40
7	V _i	1.35	1.35	1.34	1.32	1.34	1.33	1.34	1.32	1.33	1.32	1.36	1.30	*	1.31	1.31	1.30
	V _m	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.41	1.38		1.40	1.40	1.38
	C	15.5	14.5	12.9	10.5	15.5	14.0	15.0	15.9	15.5	15.9	14.5	14.0		15.0	15.0	12.5

V_i = Initial Discharge Voltage (at 30 Seconds or First Reading Taken)

V_m = Maximum Voltage Attained or Voltage at 75 Minutes

C = Capacity in Ampere Hours to 1.30 Volts End Voltage

* = Indicates Cell Failure

2

17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33
1.34	1.39	1.37	1.39	1.33	1.35	1.39	1.36	1.38	1.35	1.36	1.36	1.37	1.38	1.37	1.34	1.35
1.43	1.44	1.43	1.44	1.44	1.44	1.44	1.44	1.43	1.44	1.43	1.44	1.42	1.42	1.44	1.42	1.43
15.5	16.0	15.5	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0
1.33	1.39	1.35	1.35	1.32	1.35	1.38	1.33	1.38	1.34	1.35	1.36	1.36	1.39	1.39	1.38	1.34
1.41	1.44	1.41	1.41	1.42	1.42	1.44	1.42	1.42	1.39	1.40	1.40	1.40	1.41	1.42	1.42	1.40
1.41	1.36	1.33	1.34	1.35	1.40	1.33	1.33	1.40	1.37	1.38	1.37	1.38	1.39	1.40	1.39	1.34
1.45	1.42	1.42	1.41	1.41	1.44	1.42	1.42	1.43	1.41	1.42	1.42	1.42	1.42	1.43	1.43	1.41
1.30	1.38	1.33	1.31	1.30	1.33	1.37	1.31	1.38	1.37	1.37	1.37	1.38	1.38	1.37	1.37	1.28
1.39	1.43	1.39	1.38	1.39	1.39	1.43	1.38	1.41	1.41	1.41	1.42	1.42	1.41	1.41	1.42	1.39
1.33	1.40	1.34	1.33	1.33	1.37	1.39	1.32	1.38	1.37	1.38	1.38	1.39	1.40	1.40	1.40	1.35
1.39	1.43	1.39	1.39	1.39	1.41	1.43	1.38	1.42	1.41	1.41	1.41	1.42	1.42	1.43	1.43	1.41
1.31	1.38	1.35	1.34	1.35	1.38	1.38	1.33	1.37	1.33	1.33	1.33	1.34	1.36	1.37	1.36	1.32
1.41	1.41	1.40	1.41	1.41	1.41	1.41	1.40	1.41	1.39	1.40	1.39	1.40	1.41	1.41	1.41	1.40
15.5	14.5	14.5	14.5	14.5	14.5	14.0	15.5	15.5	16.0	16.0	16.0	16.0	15.5	15.5	15.5	15.5

50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65
1.34	1.39	1.34	1.34	1.34	1.35	1.37	1.36	1.35	1.35	1.36	1.35	1.35	1.37	1.38	1.37
1.45	1.43	1.43	1.44	1.43	1.44	1.44	1.43	1.43	1.43	1.43	1.43	1.44	1.45	1.45	1.43
15.5	16.0	16.5	16.5	16.0	16.5	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0
1.35	1.39	1.37	1.37	1.36	1.35	1.34	1.34	1.34	1.35	1.37	1.36	1.35	1.36	1.36	1.36
1.40	1.41	1.41	1.41	1.41	1.41	1.41	1.40	1.41	1.41	1.41	1.41	1.41	1.41	1.41	1.42
1.33	1.40	1.39	1.39	1.38	1.38	1.38	1.36	1.36	1.36	1.38	1.38	1.37	1.38	1.37	1.37
1.41	1.42	1.42	1.42	1.42	1.42	1.42	1.42	1.42	1.42	1.42	1.43	1.42	1.43	1.42	1.42
1.33	1.34	1.34	1.34	1.34	1.36	1.36	1.36	1.35	1.36	1.37	1.37	1.32	1.39	1.38	1.35
1.40	1.40	1.40	1.40	1.40	1.41	1.40	1.41	1.38	1.41	1.41	1.41	1.34	1.42	1.40	1.40
1.34	1.36	1.37	1.38	1.36	1.37	1.37	1.36	1.36	1.34	1.36	1.37	1.31	1.36	1.36	1.36
1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.39	1.39	1.41	1.34	1.40	1.40	1.40
1.32	1.35	1.35	1.37	1.34	1.36	1.36	1.36	1.34	1.34	1.34	1.36	1.35	1.35	1.36	1.34
1.40	1.40	1.40	1.40	1.40	1.41	1.40	1.41	1.41	1.39	1.40	1.41	1.39	1.40	1.41	1.40
1.34	1.34	1.34	1.34	1.31	1.33	1.33	1.32	1.34	1.32		1.34	1.33	1.33	1.33	1.34
1.38	1.40	1.39	1.40	1.40	1.40	1.40	1.40	1.40	1.40		1.41	1.40	1.40	1.40	1.39
12.5	14.0	14.0	14.5	14.5	14.0	13.0	12.5	13.5	14.0		13.5	14.0	13.5	14.0	13.5

a thirty day period at 90°F, this apparently results in a capacity retention on the order of one ampere hour above that displayed by cells with standard electrolyte.

An estimation of cycle life is difficult to obtain, as this characteristic is a function of discharge conditions and activated cell age among other factors. It is indicated that reliable performance might be achieved over four or five cycles during three weeks. Data also indicate that sixty-one of sixty-five cells performed satisfactorily after one month activated life including seven discharges.

TABLE NO. XXXV

EFFECT OF INITIAL DISCHARGE UPON FINAL CAPACITY
Final Capacity (Thirty Ampere Rate)

One Full Discharge		Two Full Discharges	
Al ₂ O ₃		15.6 A.H.	
No Al ₂ O ₃	15.2 A.H.	13.9 A.H.	

Cells in the initial electrolyte additive study were not fully discharged until conclusion of cycle testing, while all cells in Series No. 8 which contained Al₂O₃ additive were fully discharged twice, yielding initial as well as capacity retention data. Table No. XXXV indicates that cells which contained Al₂O₃ additive displayed less capacity immediately following activation than those with standard electrolyte. At completion of cycle and stand testing, mean cell capacity for ten cells containing Al₂O₃ additive was 15.6 A.H., or 1.7 A.H. more than for remaining cells. Cells with standard electrolyte exhibited capacity degradation during stand, while those with Al₂O₃ additive displayed increased capacity.

Another comparison relative to cells with standard electrolyte reveals that those which were initially discharged only 25%, displayed final mean capacity 1.3 A.H. greater than those initially discharged to a 1.30 cut-off voltage.

IV. SUMMARY AND CONCLUSIONS

During the extended contract period, (July 1964 - June 1965) approximately three hundred test cells were constructed with a goal of improved energy density, stand and cycle characteristics, with no sacrifice in voltage regulation or unit reliability. Although no dynamic environmental testing was included in the test program, it was intended that the final design be applicable to use in Aero-Space programs. The following is a summary of phases of study which were examined during the extended contract period.

A. Separator Evaluation

Data obtained during the original investigation period indicated impressive stand characteristics associated with multiple wraps of membranous materials in conjunction with absorbent materials next to the positive and negative. A parallel construction technique has been employed for some time in various secondary applications. Emphasis was placed upon location of efficient separator materials of decreased thickness, such that cell impedance might remain low, at the same time allowing the introduction of more active material into the cell. The separator system has been altered by replacing woven nylon with a slightly thinner matted product which is superior in electrolyte retention. Also, a thinner rayon product is used next to the negative plate. The polyamide product mentioned above might also be used next to the negative plate, with the advantage of greater stability in caustic environments. Decreases in separator thicknesses resulted in the availability of approximately 15% additional active materials.

B. Negative Formulation

Single plate studies performed upon negative plates of carefully controlled thickness revealed valuable relationships concerning apparent density.

It was indicated that zinc volume efficiency improved directly with porosity over the apparent density range investigated. Inefficiency was found to be most pronounced with apparent densities in excess of approximately 44 gm/in^3 . These data are of value not only in cell design, but in defining acceptable production materials.

C. Positive Plate Electroformation

Studies related to the positive plates were directed at achieving maximum state-of-charge, as indicated by oxygen content at termination of formation. Procedures which were investigated were 1) constant current continuous and 2) constant current interrupted.

1. Constant Current Continuous

Preliminary studies indicated that plate porosity or apparent density was by far the deciding factor in charge acceptance, as compared to electrolyte temperature, plate weight, and charge input. For this reason, later experiments were directed at investigating the combined effects of charge input and apparent density. It was confirmed that oxygen "pickup", or plate weight increase during formation, improved with densities as low as 70 gm/in^3 .

Later studies, for instance, yielded responses as great as 14.36% for 70 gm/in^3 density as compared to a maximum of 9.62% for a trial at 95 gm/in^3 density. This response is defined as (positive material weight after formation - sintered silver weight) \div sintered silver weight. A response of approximately 14.8% would correspond to the theoretical maximum, with all silver being converted to the divalent state. Final indications are that for standard formation procedures, it is advantageous to employ densities no greater than 75 gm/in^3 , with the indication that maximum state-of-charge might be achieved with a

charge input of approximately 140% of that theoretically required for complete conversion to the divalent state. These data again are invaluable not only in cell design but also for establishing process controls.

2. Constant Current Interrupted

Recognizing possible benefits of obtaining efficient plates of increased density, a brief study was conducted in an attempt to form plates of densities of 73-98 gm/in³. It was found that by interrupting the charge procedure with open circuit stand periods, charge acceptance was increased. For instance, the second such series resulted in an oxygen "pickup" of 14.58 for a plate of 80 gm/in³ density. This is comparable with the greatest response of low density trials subjected to continuous charging in earlier series. Time did not permit extensive construction of full size test cells which would reveal whether the increased density might affect discharge or stand characteristics.

D. Zinc Deposition Study

A brief study related to zinc plate formulation indicated the most favorable conditions for zinc deposition to be

- 1) plating current density of 0.65 amp/in²
- 2) 110°F bath temperature
- 3) Thirty minute plating interval (between rinse)

It was also indicated that greater control of oxide content could be obtained by employing a strong spray rinse rather than flowing water rinse.

E. Full Cell Studies

1. Ratio of Materials Study

It has been confirmed that within normal design limits, increased silver-zinc ratio favors increased initial capacity, while the reverse results in capacity characteristics more stable over a period of time. For the cycle

regime employed, it appears that a ratio of approximately 0.97:1.00 might result in nearly constant capacity over a one month period.

2. Electrolyte Additive Study

It is indicated that the addition of Al_2O_3 to the electrolyte might result in approximately one ampere hour greater capacity following thirty days activated stand.

3. Effect of Cell Tightness and Quantity of Electrolyte

Test data confirmed that neither cell group tightness or volume of electrolyte were limiting factors at their previous levels. This allowed for further minor improvements in energy density by slightly reducing the quantity of electrolyte and increasing the quantity of active materials. The ability to operate with less electrolyte apparently arises from the optimized separator system and possibly from more uniform capillarity of the active materials.

4. Final Cell Design

The following summarize the pertinent design features of the progressive cell redesign effort.

	<u>Pre-Contract</u>	<u>July 1964</u>	<u>July 1965</u>
Positive Grid	Nickel	Silver ($0.4g/in^2$)	Silver ($0.3g/in^2$)
Silver Apparent Density	$80-90\ gm/in^3$	$80\ g/in^3$	$70-75\ g/in^3$
Silver Plate Weight	$1.8 - 1.9\ gm/in^2$ AgO	$1.8 - 1.9\ gm/in^3$ AgO	$2.03\ gm/in^2$ AgO
Negative Grid	Copper ($0.25\ gm/in^2$)	Copper $0.25\ gm/in^2$	Silver $0.3\ gm/in^2$
Positive Absorbent	None	0.004" Woven Nylon	0.0025 Matted Nylon
Membrane	#133 Visking	Two Thin Cellulose	Two Thin Cellulose
Negative Absorbent	0.008" Rayon	0.0035 Rayon	0.0025 Rayon

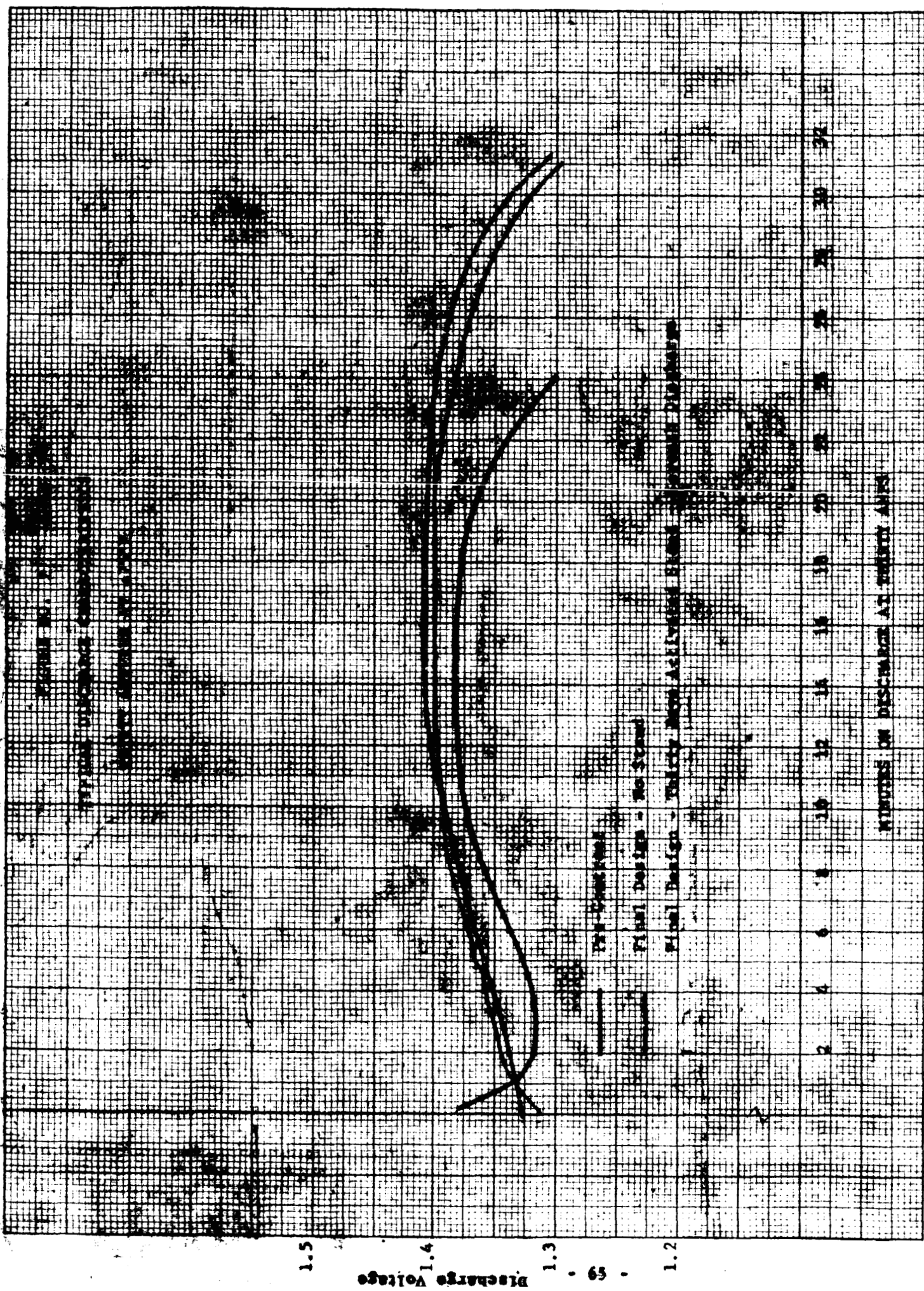
These data do not adequately reflect the improvements which were obtained in cell operation. The original cell design displayed poor recharge capabilities, being designed as a primary short stand item. Initial studies indicated that insertion of an inert material between the positive plate (a strong oxidizing agent) and the cellulosic membrane, which has long been examined with respect to attack by silver species, reduces degradation of this material. This approach represented a "marriage" of the primary and secondary systems and extended the life of the system as reported previously, while adding moderate recharge capabilities.

Replacement of nickel and copper grid materials with silver improved internal resistance characteristics and eliminated possible corrosion effects in the negative group.

Rather extensive efficiency studies relative to the positive and negative active materials resulted in greater efficiencies as well as increased appreciation of the process controls necessary to achieve these efficiencies. These alterations in plate properties apparently affected the capillary properties of the plates such that reduced quantities of absorbent materials were necessary to insure proper wetting of the cell pack. Efficiency studies also resulted in the determination of an altered ratio of materials such that more stable capacity characteristics are obtainable. Reduction of separator materials allowed the obvious increase of active materials and, therefore, cell capacity.

In summary, a cell has been designed which exhibits limited recharge capabilities, (estimated at five cycles at 25% full capacity) increased activated life, greater capacity and materials efficiency, with no known sacrifices in cell performance. Figure No. 2 compares discharge characteristics for cells at various stages of development.

10-10 TO 10-10
 7/16 X 10 INCHES
 MADE IN U.S.A.
 KEUFFEL & NAUGHTON CO.



V. REFERENCES

1. This Contract, Final Report, 31 July 1964
2. Characteristics of Separators for Alkaline Silver Oxide Zinc Secondary Batteries, Copper and Fleischer
3. AF-33(615)-1236, "Silver Migration and Transport Mechanism Studies in Silver-Zinc Batteries", Dirkse, T.P.
4. NAS 5-2860, Electric Storage Battery Company "Alkaline Battery Separator Study"
5. NAS 5-3906 The Improvement of Zinc Electrodes for Electrochemical Cells, Leeson Moos Laboratories
6. NAS 5-3873, Investigation and Improvement of Zinc Electrodes for Electrochemical Cells, Yardney Electric
7. Journal of the Electrochemical Society, Vol 117, No. 11, p. 862
8. NAS 8-5493, 31 July 1964

VI. PERSONNEL

The following number of personnel man hours were expended during the contract period.

Engineering - 6,694

Technical - 8,956.5

Total - 15,650.5

A P P E N D I X

TABLE NO. XXXVI

SILVER PLATE FORMATION STUDY

SECOND FACTORIAL EXPERIMENT

ANALYSIS OF VARIANCE

(SILVER DENSITY)

Silver Density (gm/in ³)	70	75	80	85	90	95	Total
Response	14.36	13.31	11.35	11.29	9.05	8.90	68.16
	13.49	12.75	11.99	10.63	10.64	9.62	69.12
	11.99	9.94	9.39	9.95	10.30	9.33	60.90
$\sum x_i$	39.84	36.00	32.73	31.87	29.99	27.75	198.18
\bar{x}	13.28	12.00	10.91	10.62	10.00	9.25	
$\sum(x_i^2) = 2228.92$							

$$\begin{aligned}\text{Correction Factor} &= (198.18)^2/18 \\ &= 2181.96\end{aligned}$$

$$\begin{aligned}\text{Total Sum of Squares} &= \sum(x_i^2) - \text{C.F.} \\ &= 46.96\end{aligned}$$

$$\begin{aligned}\text{Density S of S} &= (39.84^2 + \dots + (27.75^2)/3 - \text{C.F.} \\ &= 36.25\end{aligned}$$

TABLE NO. XXXVII
 SILVER PLATE FORMATION STUDY
 SECOND FACTORIAL EXPERIMENT
 ANALYSIS OF VARIANCE
 (PER CENT CHARGE)

Per Cent Charge	120	156	171	218	254	323	Total
	14.36	13.31	13.49	12.75	11.99	9.94	75.84
	11.35	11.29	11.99	10.63	9.39	9.95	64.60
	9.95	8.80	10.64	9.62	10.30	9.33	57.74
Σx_1	34.76	33.40	36.12	33.00	31.68	29.22	198.18
\bar{x}	11.59	11.13	12.04	11.00	10.56	9.74	
$\Sigma(x_1^2) = 2228.92$							

Correction Factor = 2181.96

Total Sum of Squares = 46.96

Per Cent Charge S of S = $(34.76^2 + \dots + 29.22^2)/3 - C.F.$
 = 9.67

TABLE NO. XXXVIII

SILVER PLATE FORMATION STUDY

SECOND FACTORIAL EXPERIMENT

Silver Density	Variance $\frac{\sum x_i^2 - (\sum x_i)^2}{9}$	Response Mean
70 gm/in ³	2.88	13.28
75	6.52	12.00
80	3.68	10.91
85	0.90	10.62
90	1.40	10.00
95	0.34	9.25

TABLE NO. XXXIX

SILVER PLATE FORMATION STUDY

SECOND FACTORIAL EXPERIMENT

CALCULATION OF VARIANCE

Per Cent Charge	Variance	Response Mean
120	14.18	11.59
156	11.08	11.13
171	4.07	12.04
218	5.10	11.00
254	3.48	10.56
323	0.26	9.74

TABLE NO. XL

SILVER PLATE FORMATION STUDY

ANALYSIS BY COMPARISON OF MEANS

Silver Density	70	75	80	85	90	95 gm/in ³
% Gain	14.36	13.31	11.35	11.29	9.05	8.80
	13.49	12.75	11.99	10.63	10.64	9.62
	11.99	9.94	9.37	9.95	10.30	9.33
	39.84	36.00	20.73	31.87	29.99	27.75
x	13.28	12.00	10.91	10.62	10.00	9.25

Overall Mean = 11.01

TABLE NO. XLI

SILVER PLATE FORMATION STUDY

ANALYSIS BY COMPARISON OF MEANS

Per Cent Charge	120	156	171	218	254	323
% Gain	14.36	13.31	13.47	12.75	11.99	9.94
	11.35	11.29	11.99	10.63	9.39	9.95
	9.05	8.80	10.64	9.62	10.30	9.33
Σx	34.76	33.40	36.12	33.00	31.68	29.22
\bar{x}	11.59	11.33	12.04	11.00	10.56	9.92

Overall Mean = 11.07

TABLE NO. XLII

SILVER PLATE FORMATION STUDY

THIRD FACTORIAL EXPERIMENT

ANALYSIS OF VARIANCE

(ARRANGED BY SILVER DENSITY)

Silver Density	L 70	M 75	H 80	Total
Per Cent Weight Gain	12.97	13.07	12.28	38.32
Per Cent Weight Gain	14.15	14.03	12.33	40.51
Per Cent Weight Gain	12.28	13.28	12.48	38.04
Per Cent Weight Gain	13.40	12.16	10.70	36.26
Per Cent Weight Gain	14.57	13.98	12.54	41.09
Per Cent Weight Gain	13.76	11.67	11.82	37.25
ΣXi	81.13	78.19	72.15	231.47
\bar{X}	13.52	13.03	12.03	
$(\Sigma Xi)^2$	1100.42	1023.52	870.03	2994.11

$$\begin{aligned}
 \text{Correction Factor} &= (231.49)^2 \div 18 \\
 &= 53578.36 \div 18 \\
 &= 2976.58
 \end{aligned}$$

$$\begin{aligned}
 \text{Total Sum of Squares} &= (\text{Total, each response})^2 - CF \\
 &= 2994.11 - 2976.58 \\
 &= 17.53
 \end{aligned}$$

$$\begin{aligned}
 \text{Density Sum of Squares} &= \frac{(81.19)^2 + (78.19)^2 + (72.20)^2}{6} - 2976.58 \\
 &= \frac{17902.10}{6} - 2976.58 \\
 &= 2983.68 - 2976.58 \\
 &= 7.10
 \end{aligned}$$

TABLE NO. XLIII

SILVER PLATE FORMATION STUDY

THIRD FACTORIAL EXPERIMENT

ANALYSIS OF VARIANCE (PER CENT CHARGE)

Per Cent Charge	L 120	M 140	H 160	Total
Per Cent Gain	12.97	14.15	12.28	39.40
Per Cent Gain	13.07	14.03	13.28	40.38
Per Cent Gain	12.28	12.33	12.48	37.09
Per Cent Gain	13.40	14.57	13.76	41.73
Per Cent Gain	12.16	13.98	11.67	37.81
Per Cent Gain	10.70	12.54	11.82	35.06
ΣX_i	74.58	81.60	75.29	231.47
\bar{X}	12.43	13.60	12.54	
$(\Sigma X_i)^2$	931.76	1114.07	948.15	2994.11

$$\begin{aligned}
 \text{Correction Factor} &= (231.47)^2 \div 18 \\
 &= 53578.36 \div 18 \\
 &= 2976.58
 \end{aligned}$$

$$\begin{aligned}
 \text{Total Sum of Squares} &= (\text{Total, each response})^2 - CF \\
 &= 2994.11 - 2976.58 \\
 &= 17.53
 \end{aligned}$$

$$\begin{aligned}
 \% \text{ of Charge Sum of Squares} &= (74.58)^2 + (81.60)^2 + (75.29)^2 - CF \\
 &= \frac{17889.3205}{6} - 2976.58 \\
 &= 2981.55 - 2976.58 \\
 &= 4.97
 \end{aligned}$$

TABLE NO. XLIV

SILVER PLATE FORMATION STUDY

THIRD FACTORIAL EXPERIMENT

CALCULATIONS OF VARIANCE FOR EACH

SILVER DENSITY

$$\text{Variance} = \sum X_i - \frac{(\sum X_i)^2}{n}$$

$$\begin{aligned}\text{Variance, Density 70} &= 1100.42 - \frac{6582.08}{6} \\ &= 1100.42 - 1097.01 \\ &= 3.41; \bar{X} = 13.52\end{aligned}$$

$$\begin{aligned}\text{Variance, Density 75} &= 1023.52 - \frac{6113.68}{6} \\ &= 1023.52 - 1018.95 \\ &= 4.57; \bar{X} = 11.67\end{aligned}$$

$$\begin{aligned}\text{Variance, Density 80} &= 870.03 - \frac{5205.62}{6} \\ &= 870.03 - 867.60 \\ &= 2.43; \bar{X} = 12.03\end{aligned}$$

TABLE NO. XLV

SILVER PLATE FORMATION STUDY

THIRD FACTORIAL EXPERIMENT

CALCULATIONS OF VARIANCE FOR EACH

PER CENT OF CHARGE

$$\text{Variance} = \sum X_i^2 - \frac{(\sum X_i)^2}{n}$$

$$\text{Variance, 120\% Charge} = 931.76 - \frac{(74.58)^2}{6}$$

$$= 931.76 - 927.03$$

$$= 4.73; \bar{X} = 12.43$$

$$\text{Variance, 140\% Charge} = 1114.07 - \frac{(81.60)^2}{6}$$

$$= 1114.07 - \frac{6658.66}{6} = 1114.07 - 1109.76$$

$$= 4.31; \bar{X} = 13.60$$

$$\text{Variance, 160\% Charge} = 948.12 - \frac{(75.29)^2}{6}$$

$$= 948.15 - \frac{(75.29)^2}{6}$$

$$= 948.15 - \frac{5668.58}{6}$$

$$= 948.15 - 944.76$$

$$= 3.39; \bar{X} = 12.54$$

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